EPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC SUPERFUND METHODS

Multi-Media, Multi-Concentration

ISM02.4
October 2016

STATEMENT OF WORK

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INORGANIC ABBREVIATIONS/ACRONYM LIST	
ABBREVIATION/ACRONYM	DEFINITION
AA	Atomic Absorption
ASB	Analytical Services Branch
ASB CLP COR	Analytical Services Branch Contract Laboratory Program Contracting Officer's Representative
°C	Degrees Celsius (unit of measurement)
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCS	Contract Compliance Screening
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLP	EPA Contract Laboratory Program
CO	Contracting Officer
COC	Chain of Custody
COR	Contracting Officer's Representative
CRQL	Contract Required Quantitation Limit
CSF	Complete SDG File
CVAA	Cold Vapor Atomic Absorption Spectroscopy
%D	Percent Difference
DF	Dilution Factor
DRD	Data Receipt Date
DTD	Document Type Definition
Dup	Duplicate Sample
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
EXES	Electronic Data Exchange and Evaluation System
FCC	Federal Communications Commission
FEP	Fluorinated Ethylene Propylene
g	Gram (unit of measurement)
HRS	Hazard Ranking System
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectrometer or Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometer or Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICSA	Interference Check Sample Solution A
ICSAB	Interference Check Sample Solution AB
ID	Identifier
IEC	Interelement Correction
ICV	Initial Calibration Verification
IPC	Instrument Performance Check
IR	Infrared
kg	Kilogram (unit of measurement)
L	Liter (unit of measurement)
Lab	Laboratory
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
LRD	Laboratory Receipt Date

INORGANIC ABBREVIATIONS/ACRONYM LIST	
ABBREVIATION/ACRONYM	DEFINITION
MA	Modified Analysis
MDL	Method Detection Limit
mg	Milligram (unit of measurement)
mL	Milliliter (unit of measurement)
mm	Millimeter (unit of measurement)
MS	Matrix Spike
MSDS	Material Safety Data Sheet
NCS	Non-Client Sample
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
nm	Nanometer (unit of measurement)
NSCEP	National Service Center for Environmental Publications
OSHA	Occupational Safety and Health Administration
OSRTI	EPA Office of Superfund Remediation and Technology Innovation
PB	Preparation Blank
PDF	Portable Document Format
PDS	Post-Digestion/Distillation Spike
PE	Performance Evaluation
PRPs	Potentially Responsible Parties
PT	Proficiency Testing
PTFE	Polytetrafluoroethylene
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
QMP	Quality Management Plan
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
RPD	Relative Percent Difference
%S	Percent Solids
SA	Spike Added
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	Serial Dilution
SD	Standard Deviation
SDG	Sample Delivery Group
SEDD	Staged Electronic Data Deliverable
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SPLP	Synthetic Precipitation Leaching Procedure
SR	Sample Result
SSR	Spiked Sample Result
TAL	Target Analyte List
TCLP	Toxicity Characteristic Leaching Procedure
TR	Traffic Report
TR/COC	Traffic Report/Chain of Custody
µg	Microgram (unit of measurement)
UTF-8	Unicode Transformation Format - 8 bit
VTSR	Validated Time of Sample Receipt
W3C	World Wide Web Consortium
XML	eXtensible Markup Language

EXHIBIT A

SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

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1.0   PURPOSE

The purpose of this analytical service is to provide analytical data
for use by the U.S. Environmental Protection Agency (EPA), in support
of the investigation and clean-up activities under the Comprehensive
Environmental Response, Compensation, and Liability Act of 1980
(CERCLA) and the Superfund Amendments and Reauthorization Act of 1986
(SARA). Other EPA Program Offices, as well as customers outside the
Agency, that have similar analytical data needs also use this service.

2.0   DESCRIPTION OF SERVICE

This Statement of Work (SOW) provides a contractual framework for
laboratories to perform analytical services. This framework applies
EPA Contract Laboratory Program (CLP) analytical methods for the
isolation, detection, and quantitative measurement of 23 metals and
cyanide in aqueous/water and soil/sediment samples, and total metals
analysis in wipes. The SOW also includes Toxicity Characteristic
Leaching Procedure (TCLP) and Synthetic Precipitation Leaching
Procedure (SPLP) leachate extraction procedures. The analytical
service contract provides the methods to be used and the specific
contractual requirements by which the EPA will evaluate the data.

3.0   DATA USES

This analytical service provides data used for a variety of purposes,
such as: determining the nature and extent of contamination at a
hazardous waste site, assessing priorities for response based on risks
to human health and the environment, determining appropriate clean-up
actions, and determining when remedial actions are complete. The data
may be used in all stages in the investigation of hazardous waste
sites, including site inspections, Hazard Ranking System (HRS) scoring,
remedial investigation/feasibility studies, remedial design,
treatability studies, and removal actions.

In addition, the Contractor must be aware of the importance of
maintaining the integrity of data generated under the contract, since
it is used to make major decisions regarding public health and
environmental welfare. The data may also be used in litigation against
Potentially Responsible Parties (PRPs) in the enforcement of Superfund
legislation.

4.0   SUMMARY OF REQUIREMENTS

The SOW is comprised of eight exhibits:

- Exhibit A – Summary of Requirements

- Exhibit B – Reporting and Deliverables Requirements

- Exhibit C – Inorganic Target Analyte List and Contract Required
  Quantitation Limits

- Exhibit D – Analytical Methods

- Exhibit E – Quality Systems

- Exhibit F – Programmatic Quality Assurance/Quality Control Elements

- Exhibit G – Glossary of Terms

- Exhibit H – Format for Electronic Data Deliverables

4.1   Major Task Areas

For each sample, the Contractor shall perform the tasks described in
each section. Specific requirements for each task are detailed in the
exhibits referenced.

4.1.1   Sample Receiving, Storage, and Disposal

The Contractor will receive samples from potential hazardous waste
sites and shall store and maintain these samples under proper chain
of custody (COC) procedures. The Contractor shall follow procedures
outlined in Section 5.0 of this Exhibit for proper sample receipt
and handling as well as each Exhibit D – Analytical Methods for
proper storage and disposal of unused portion of samples. All
anomalies and identified issues shall be communicated to the EPA via
the CLP Sample Management Office (SMO) Contractor.

4.1.2   Sample Preparation and Analysis

The Contractor is advised that the samples received under this
contract are usually from known or suspected hazardous waste sites
and may contain high levels of organic and inorganic materials of a
potentially hazardous nature and of unknown structure and
concentration, and should be handled throughout the analysis with
appropriate caution. It is the Contractor's responsibility to take
all necessary measures to ensure laboratory safety.

4.1.2.1   The Contractor shall prepare samples as described in the
            respective Exhibit D – Analytical Methods for the requested
            analysis type. Sample preparation methods shall remain
            consistent for all samples analyzed within a Sample Delivery
            Group (SDG).

4.1.3   Sample Reporting and Resubmission of Data

4.1.3.1   Required formats for the reporting of data are found in Exhibit
            B – Reporting and Deliverables Requirements and Exhibit H –
            Format for Electronic Data Deliverables. The Contractor shall be
            responsible for completing and submitting analysis data sheets
            and electronic data as requested in a format specified in this
            SOW and within the time specified in Exhibit B – Reporting and
            Deliverables Requirements, Section 1.1.

4.1.3.2   Use of formats other than those approved will be deemed as
            noncompliant. Such data are unacceptable. Resubmission in the
            specified format will be required at no additional cost to the
            Government.

4.1.4   Quality Assurance/Quality Control

The Contractor shall maintain a Quality Assurance Project Plan
(QAPP) with the objective of providing sound analytical chemical
measurements. This program shall incorporate the Quality Control
(QC) procedures, any necessary corrective action, and all
documentation required during data collection, as well as the
Quality Assurance (QA) measures performed by management to ensure
acceptable data production.

4.1.4.1   The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D – Analytical Methods and F – Programmatic Quality Assurance/Quality Control Elements. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit E – Quality Systems, and shall be reported in accordance with Exhibit B – Reporting and Deliverables Requirements and Exhibit H – Format for Electronic Data Deliverables.

4.1.4.2   Additional QC shall be conducted in the form of the analysis of Performance Evaluation (PE) samples submitted to the laboratory by the EPA. Unacceptable results of all such QC or PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to the EPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by the EPA, as well as meeting the contract requirements for analysis (Exhibit D – Analytical Methods); QA/QC (Exhibit F – Programmatic Quality Assurance/Quality Control Elements); data reporting and other deliverables (Exhibits B – Reporting and Deliverables Requirements and H – Format for Electronic Data Deliverables); and sample custody, sample documentation, and Standard Operating Procedure (SOP) documentation (Exhibit E – Quality Systems). As an alternative to data rejection, the EPA may require reanalysis of noncompliant samples. Reanalysis will be performed by the Contractor at no additional cost to the EPA.

4.1.5   Modified Analysis

The Contractor may be requested by the EPA to perform a Modified Analysis (MA). The modifications may include, but are not limited to: modified preparation or analysis procedures; additional analytes; sample matrices other than those present in the SOW; and/or lower quantitation limits. The requests will be made in writing, prior to sample scheduling. All contract requirements specified in the SOW/Specifications will remain in effect unless specifically modified.

5.0   SAMPLE RECEIPT AND HANDLING

5.1   Chain of Custody

The Contractor shall receive and maintain samples under proper COC procedures. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete SDG File (CSF) (See Exhibit B – Reporting and Deliverables Requirements). The Contractor shall establish and use appropriate procedures to handle confidential information received from the EPA.

5.2   Sample Scheduling

5.2.1   Sample shipments to the Contractor's facility will be scheduled and coordinated by the CLP SMO. The EPA may request analyses that include all or a subset of the Target Analytes listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits. The EPA may also request modified analyses due to the nature of the samples or project requirements. The Contractor shall communicate with SMO personnel as necessary, throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

5.2.2   The Contractor shall accept all samples scheduled by SMO, provided that the total number of samples received in any calendar month does not exceed the monthly limitation defined in the contract. Should the Contractor elect to accept additional samples, the Contractor shall remain bound by all contract requirements for analysis of those samples accepted.

5.3   Sample Shipments

5.3.1   Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive sample shipments at any time the delivery service is operating, including weekends.

5.3.2   Unless otherwise instructed by the EPA Region or originating sampler, the Contractor shall be required to routinely return sample shipping containers to the appropriate sampling office within 14 calendar days following shipment receipt. This shipment must be done via ground transportation only pending receipt of a valid return authorization, unless specifically instructed to do otherwise. The Contractor will be provided a shipping mechanism by the EPA Region or originating sampler (e.g., field sampler). The Contractor shall ensure that the account numbers provided are used only for the return of Government-owned shipping containers.

5.3.2.1   The Contractor shall remove packing and other materials from the shipping containers before each pick-up and shall ensure that the shipping containers are clean. The Contractor can determine from visual inspection whether the shipping container is clean.

5.4   Sample Receipt

5.4.1   If insufficient sample amount (less than the required amount) is received to perform the analyses, the Contractor shall notify SMO and proceed with the analysis of the sample at reduced volume. The Contractor shall document this issue in the SDG Narrative.

5.4.2   If the Contractor receives broken sample containers, with enough remaining sample to perform sample analysis, but potentially not enough volume to analyze any possible re-extractions/reanalyses, the Contractor shall note the issue in the SDG Narrative, proceed with analysis of the samples, and notify SMO. If re-extraction/ reanalyses are necessary, the Contractor shall contact SMO. The Contractor shall document the provided resolution in the SDG Narrative.

5.4.3   If the Contractor encounters other problems with samples or related documentation [e.g., mixed media, sample pH, sample documentation and paperwork such as Traffic Report/Chain of Custody (TR/COC) Records not with shipment, sample and TR/COC Record do not correspond], the Contractor shall immediately contact SMO for resolution.

5.4.4   Shipping Container Temperature Monitoring

5.4.4.1   To monitor the temperature of the sample shipping container more effectively, a sample shipping container temperature indicator bottle may be included with each shipping container shipped. The applicable temperature blank will be clearly labeled.

5.4.4.2   When a shipping container temperature indicator bottle is included in the sample shipping container, the Contractor shall use the supplied shipping container temperature indicator bottle to determine the shipping container temperature. The temperature of the sample shipping container shall be measured and recorded immediately upon opening the shipping container, and prior to unpacking the samples or removing the packing material.

5.4.4.3   To determine the temperature of the shipping container, the Contractor shall locate the shipping container temperature indicator bottle in the sample shipping container, invert it several times, remove the cap, and insert a calibrated [National Institute of Standards and Technology (NIST)-traceable] thermometer into the shipping container temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the thermometer used shall be capable of measuring and registering the temperature of the shipping container with an accuracy of ±1°C.

5.4.4.4   If a temperature indicator bottle is not present in the shipping container, an alternative means of determining shipping container temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining shipping container temperature. Other devices (e.g., infrared thermometer) which can measure temperature may be used if they can be calibrated to ±1°C.

5.4.4.5   If a temperature indicator bottle is not present in the shipping container, and the temperature of the shipping container is not less than or equal to 6°C, the Contractor shall note the issue, and the method used to determine the temperature, in the SDG Narrative and proceed with analysis of the samples. If the temperature exceeds 10°C and the samples are soil/sediment samples for any analytical method or aqueous/water samples for cyanide analysis, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the EPA for instructions on how to proceed. SMO will in turn notify the Contractor of the EPA's decision. The Contractor shall document the EPA's decision and the EPA Sample Numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.

5.4.4.6   Liquid bearing thermometers such as mercury or alcohol thermometers shall be traceable to NIST calibration and verified at least annually, and whenever the thermometer has been exposed to temperature extremes. The correction factor shall be indicated on the thermometer, and the date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection. The NIST thermometer shall be recalibrated at least every five years or whenever the thermometer has been exposed to temperature extremes.

Digital thermometers, thermocouples, and other similar electronic temperature measuring devices shall be calibrated at least quarterly. The date the thermometer was calibrated and the calibration factor shall be kept as prescribed in the laboratory's QA documents and be available for inspection.

When an infrared (IR) detection device is used to measure the temperature of samples, the device shall be verified at least every six months using an NIST certified thermometer over the full temperature range that the IR thermometer will be used. This would include ambient (20-30°C), iced (4°C), and frozen (0 to -5°C). Each day of use, a single check of the IR shall be made by measuring the temperature of a bottle of water, that contains a calibrated thermometer, at the temperature of interest. Agreement between the two readings should be within 0.5°C, or the device shall be recalibrated. The daily checks of the IR shall be documented and the records maintained on file.

5.4.5   Recording Sample pH

5.4.5.1   The pH for all aqueous/water samples received by the Contractor shall be measured, using a method capable of demonstrating that proper preservation was performed (e.g., pH test strips, electronic hand-held pen, pH meter), and recorded. The pH shall be determined using a small aliquot of the sample to prevent contamination. Under no circumstances shall a strip or any device be inserted into a sample bottle for the purpose of determining pH.

5.4.5.2   All pens and pH meter electrodes shall be rinsed with reagent water between sample readings.

5.5   Sample Case

Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case Number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.

5.5.1   A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or

- Each 20 samples (excluding PE samples) within a Case; or

- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with receipt of the first sample in the SDG).

- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining an SDG.

- All samples scheduled with the same level of deliverables.

5.5.2   Samples may be assigned to SDGs by matrix (i.e., all soil/sediment in one SDG, all aqueous/water in another), at the discretion of the laboratory. If PE samples are received within a Case, they shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received and shall <u>not</u> be made retroactively. The SDG may exceed the 20 samples limit since the limitation excludes PE samples.

5.5.3   Each sample received by the Contractor will be labeled with an EPA Sample Number and accompanied by a TR/COC Record bearing the Sample Number and descriptive information regarding the sample. The EPA Sample Numbers are continuous, without spaces or hyphens. If the sample numbers do not conform to this requirement, contact SMO. The Contractor shall complete and sign the TR/COC Record, recording the date of sample receipt and sample condition on receipt for each sample container.

5.5.3.1   The Contractor shall follow the instructions given on the TR/COC Record in choosing the QC samples, when such information is provided. If no QC sample is designated on the TR/COC Record, the Contractor shall select a sample and notify SMO for EPA Regional acceptance. SMO shall contact the EPA Region for confirmation immediately after notification.

5.5.3.2   If the Sampler designated two (or more) samples as QC for the same matrix, and the QC samples are not specifically labeled with the analysis they are to be used for (dissolved metals and total metals), then the Contractor is to contact SMO to report the issue. SMO shall then contact the EPA Region and notify the Contractor of the EPA Regional decision. If the Sampler did not designate QC samples, then the Contractor is to select a sample for QC and to contact SMO to report the issue.

5.5.4   The date of delivery of the SDG, or any samples within the SDG, is the date that the last sample in the SDG is received. Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample TR/COC Record.

5.5.5   The Contractor shall submit electronic copy(ies) of signed TR/COC Record as Portable Document Format (PDF) file(s) for all samples in an SDG to SMO via the Superfund Analytical Services SMO Portal at https://epasmoweb.fedcsc.com within 3 working days following the receipt of the last sample in the SDG. TR/COC Records shall be submitted with their SDG information as specified in Exhibit B – Reporting and Deliverables Requirements.

5.5.6   The EPA Case Numbers, SDG Numbers, and EPA Sample Numbers shall be used by the Contractor in identifying samples received under this contract, both verbally and in reports/correspondence.

5.5.7   The Contractor shall immediately notify SMO regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.

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EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B – Reporting and Deliverables Requirements

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1.0   CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1   Report Deliverable Schedule

The following table identifies the contract reporting and deliverables requirements, and specifies the distribution that is required for each deliverable.

TABLE 1. DELIVERABLE SCHEDULE

Item		No. of Copies[1]	Delivery Schedule	Distribution		
				SMO	Region	QATS
A.	Sample Traffic Reports/Chain of Custody (TR/COC) Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG).	X		
B.[2,3]	Complete SDG File (CSF)	1	XX[4] days after Validated Time of Sample Receipt (VTSR) of last sample in SDG.		X	
C.[2,5,8]	Copy of CSF and Hardcopy Data in Portable Document Format (PDF) Format	1	XX[4] days after VTSR of last sample in SDG.	X		
D.[2,6]	Preliminary Results	1	Within 48 hours after receipt of each sample at laboratory, if requested.	X	X	
E.[2,8]	Electronic Data Deliverable (EDD)	1	XX[4] days after VTSR of last sample in SDG.	X		
F.[2]	Proficiency Testing (PT) Audits	1	XX[4] days after VTSR of last sample in SDG.	X		
G.[7,8]	Determination of Method Detection Limits (MDL) And Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) Interelement Correction (IEC) Factors	1	MDL values in spreadsheet format specified in Appendix A of Exhibit H prior to analysis of field samples, annually thereafter, and after major instrument adjustments to SMO and QATS. MDL and IEC study data prior to analysis of field samples, annually thereafter, and after major instrument adjustments to QATS only. Submission of all deliverables within 7 days of determinations.	X		X

TABLE 1. DELIVERABLE SCHEDULE (CON'T)

	Item	No. of Copies[1]	Delivery Schedule	Distribution		
				SMO	Region	QATS
H.	Standard Operating Procedures (SOPs)	1	Submit within 60 days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 4.0) Submit amended documents within 14 days of amended SOP(s) as directed in Exhibit E, Section 4.4.			X
I.	Quality Assurance Project Plan (QAPP)	1	Submit within XX[4] days after contract award. Submit the latest version within 7 days of receipt of written request, to recipients as directed. (See Exhibit E, Section 3.0) Submit amended documents within 14 days of amended QAPP as directed in Exhibit E, Section 3.3.			X
J.	Instrument Electronic Data	Lot	Retain for 3 years after data submission of the reconciled CSF. Submit within 7 days of receipt of written request, to recipients as directed. (See Exhibit F, Section 8.3)	As Directed		X
K.	Digestates	Lot	Retain for total metals (excluding mercury) 180 days after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		
L.	Samples	Lot	Retain for 60 days after data submission. Submit within 7 days after receipt of written request, to recipients as directed.	As Directed		

Footnotes:

[1] The number of copies specified is the number of copies required to be delivered to each recipient.

[2] **DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE.** Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date that all samples have been delivered. **If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.**

[3] CSF will contain the original Sample Data for Level 2a, 2b, and 3 deliverables, plus all of the original documents described in Exhibit B, Section 2.4.

[4] The number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.

[5] Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the U.S. Environmental Protection Agency's Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR) and Analytical Services Branch CLP COR (ASB CLP COR). Supplemental data (i.e., logbooks) may be requested in writing from the EPA Regional staff or the ASB CLP COR. All written communication sent by the EPA must include the EPA Regional CLP COR in the distribution list. If the EPA Regional CLP COR has not been included in the distribution list, contact the ASB CLP COR.

[6] If requested at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of Form 1-IN sample analyses and field Quality Control (QC) analyses. The Contractor shall provide the SMO copy via the EPA Electronic Data Exchange and Evaluation System (EXES) at https://epasmoweb.fedcsc.com as a PDF file as preliminary results. The PDF file name should be PR_Case Number_SDG Number_Contract Number_Method. Sample TR/COC Records and SDG Cover Page (per Exhibit B, Section 2.7.1) shall be submitted with the Preliminary Results. The designated Regional recipient shall receive the Preliminary Results as a PDF file or in alternative electronic formats (e.g., Microsoft® Word) via email. The Contractor will be notified of the email address and format at the time of sample scheduling.

   NOTE: Preliminary Results Delivery Schedule:

       If a sample requiring Preliminary Results arrives at the laboratory before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received at the laboratory after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

[7] Results required in each CSF.

[8] The Contractor shall provide SMO the electronic files via EXES at https://epasmoweb.fedcsc.com.

1.2   Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1 – Deliverable Schedule.

Sample Management Office (SMO)[1]:

> Delivery instructions shall be provided upon contract award.

EPA Region:

> SMO will provide the Contractor with the list of addressees for data delivery for the 10 EPA Regions. SMO will provide the Contractor with updated EPA Regional address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-case basis.

EPA Regional CLP Contracting Officer's Representative:

> SMO will provide the Contractor with the list of addresses for the EPA Regional CLP CORs. SMO will provide the Contractor with updated name/address lists as necessary throughout the period of the contract.

Quality Assurance Technical Support (QATS)[2]:

> Delivery instructions shall be provided upon contract award.

2.0   REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

2.1   Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1 (for hardcopy) and Exhibit H (for electronic). The required content and form of each deliverable are described in this Exhibit. All reports and documentation **shall be**:

- Legible;

- Clearly labeled and completed in accordance with instructions in this Exhibit;

- Arranged in the order specified in this Exhibit;

- Paginated sequentially according to instructions in this Exhibit; and

- Double-sided.

- Information reported on the forms listed in this Exhibit [excluding the Sample Log-In Sheet (DC-1) and the Complete SDG File (CSF) Inventory Sheet (DC-2)] must be computer-generated.

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[1] SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by the EPA.
[2] QATS is a Contractor-operated facility operating under the QATS contract awarded and administered by the EPA.

- The Contractor shall use EPA Case Numbers, SDG Numbers, and EPA Sample Numbers to identify samples received under this contract, verbally, electronically, and in reports and correspondence. The Contract Number and the Statement of Work (SOW) Number shall be specified in all correspondence. The Modification Analysis Number (MA No.) shall also be included for all Modified Analyses.

2.1.1   The Contractor shall submit Staged Electronic Data Deliverable (SEDD) Level 2a, Level 2b, or Level 3 deliverables as specified at the time of scheduling.

- Level 2a deliverables consist of a specified limited subset of the data reporting forms as specified in this Exhibit.

- Level 2b deliverables include all data reporting forms as specified in this Exhibit.

- Level 3 deliverables include all data reporting forms and supporting raw data as specified in this Exhibit.

2.1.2   Section 3.0 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide the EPA with all required data. Section 4.0 of this Exhibit contains the required Data Reporting Forms in Agency-specified format. Data elements and instructions for electronically reporting data are contained in Exhibit H – Format for Electronic Data Deliverables.

2.2   Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected, at no additional cost to the EPA.

2.2.1   Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through an EPA Regional CLP COR action, or through an EPA Regional data reviewer's request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients (SMO and EPA Region) and to the EPA's designated recipient when a written request for a copy of the CSF has been made within 5 business days (3 business days for a 7-day turnaround) of receipt of the request. A cover letter shall be included which describes what data are being delivered, to which EPA Case Number(s) and SDG Number(s) the data pertains, and who requested the data. Corrected data submitted as "Additional Data" at the request of an EPA Regional data reviewer shall only include the affected pages and be accompanied by a revised SDG Narrative (described in Section 2.4.5 of this Exhibit) documenting the reason(s) for the resubmittal.

2.2.2   Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to both contractual data recipients (SMO and EPA Region), and to the EPA's designated recipient when a written request for a copy of the CSF has been made, within 6 business days of receipt of the request. In all instances, the Contractor shall include a cover sheet (Laboratory Response to Results of Contract Compliance Screening). Electronic deliverables shall be submitted or resubmitted to SMO only. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the EPA Region.

2.3   Sample Traffic Report/Chain of Custody Records

2.3.1   Each sample received by the Contractor shall be labeled with an EPA
        Sample Number and will be accompanied by a TR/COC Record bearing the
        Sample Number and descriptive information regarding the sample. The
        Contractor shall complete the TR/COC Record, recording the date of
        sample receipt, verifying the number of samples, and signing the
        TR/COC Record.

2.3.1.1 Upon receipt, the Contractor shall sign for the receipt of
        samples in the COC Record section. The laboratory Sample
        Custodian or designated recipient opening and verifying the
        contents of the shipping container shall then verify receipt of
        all samples identified within the CLP Traffic Report section and
        sign and date the signature box located in the CLP Traffic Report
        section. If a non-CLP TR/COC Record is submitted with the
        samples (e.g., a Regional TR/COC Record), then the Contractor
        shall: (1) record the receipt date of the samples and sign the
        TR/COC Record to maintain the chain-of-custody, and (2) the
        Sample Custodian or designated recipient shall sign and date the
        TR/COC Record to verify sample information.

        NOTE: If the laboratory is requested to transfer samples to
              another facility, the Contractor shall date and enter the
              name of the facility to where the samples will be
              transferred on the CLP TR/COC Record and document in the
              SDG Narrative.

2.3.1.2 The Contractor shall also enter the SDG Number, Case Number, and
        the Laboratory Contract Number on the CLP TR/COC Record. The EPA
        Sample Number of the first sample received in the SDG is the SDG
        Number. When several samples are received together in the first
        SDG shipment, the SDG Number shall be the lowest sample number
        (considering both alpha and numeric designations) in the first
        group of samples received under the SDG. Under no circumstances
        should any SDG Number be replicated within a Case. If necessary,
        select an alternative sample number for the SDG Number. The SDG
        Number is also reported on all data reporting forms (see Exhibit
        B, Section 3.0 - Form Instructions).

2.3.2   The Contractor shall submit TR/COC Records in SDG sets (i.e., TR/COC
        Records for all samples in an SDG), with an SDG Definition Sheet
        attached. The SDG Definition Sheet shall contain the following
        items:

        • Laboratory Name;

        • Contract Number;

        • Modified Analysis Number (if applicable);

        • Case Number;

        • List of the method/analysis for each sample; and

        • List of EPA Sample Numbers of all samples in the SDG, identifying
          the first and last samples received, and their Laboratory Receipt
          Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations).

2.3.3 EPA Sample Numbers are continuous, without spaces or hyphens. The original Sample TR/COC Record page, with laboratory receipt information and signed with an original Contractor signature, shall be submitted for each sample in the SDG.

2.3.4 If samples are received at the laboratory with multi-sample TR/COC Records, all the samples on one multi-sample TR/COC Record may not necessarily be in the same SDG. In this instance, the Contractor must make the appropriate number of photocopies of the TR/COC Record and submit one copy with each SDG Definition Sheet.

2.4 Complete Sample Delivery Group File

The CSF is described in this section. Sections 2.4.7 through 2.4.10 are specific to the individual analytical methods. If analysis by one or more of the analytical methods is not required, then those method sections are not required as a deliverable. Each method section shall include data for analysis of all samples in one SDG, including field samples, calibrations, QC samples, and supporting documentation. The CSF shall be complete before submission. The CSF shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package).

2.4.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to the EPA with another Case/SDG. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3.0 and 4.0; and organized according to Form DC-2.

NOTE: The Contractor shall retain a legible electronic (PDF) or hardcopy of the CSF for 365 days after submission of the reconciled data package to the Government. After this time, the Contractor may dispose of the package.

2.4.2 The CSF shall consist of the following original documents:

- Completed SDG Cover Page with signature and date

- EPA Sample TR/COC Record

- Completed and signed Sample Log-In Sheet [Form DC-1]

- Completed and signed Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

- SDG Narrative

- All original shipping documents, including, but not limited to, the following documents:

  o Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information);

  o Sample Tags (if present) sealed in plastic bags; and

　　　　o All original receiving documents, including, but not limited to, other receiving forms or copies of receiving logbooks.

　　　　NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to the EPA, as well as copies that are altered in any fashion, are also deliverables to the EPA. Send the original to the EPA Region and a copy to SMO. Send to the EPA's designated recipient only upon written request.

2.4.3　For Level 3 deliverables, all original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:

- Percent Solids Log;

- Original preparation and analysis forms, or copies of preparation and analysis logbook pages;

- Internal sample and sample digestate and distillate transfer Chain of Custody Records; and

- Performance Evaluation (PE) Instruction forms.

2.4.4　All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:

- Communication logs;

- Copies of personal logbook pages;

- All handwritten SDG-specific notes; and

- Any other SDG-specific documents not covered by the above.

If the Contractor does submit SDG-specific documents to the EPA after the submission of the CSF, the documents shall be identified with submission codes. For example, if a page or pages were submitted with errors, the corrected pages would be identified with the Case and SDG Number, and the code R#, where the "#" is incremented for any subsequent resubmissions. If a page has been left out of a CSF, it must be submitted with the code A#. If the entire CSF is to be resubmitted, it must be designated with the code RS#. A revised Form DC-2 should be submitted, and the submission codes and locations of the documents in the CSF shall be recorded in the "Other Records" section on the revised Form DC-2.

2.4.5　SDG Narrative

This document shall be clearly labeled "SDG Narrative" and shall contain: Laboratory Name, SOW Number, Contract Number, Case Number, SDG Number, Modified Analysis Number (if applicable), and detailed documentation of any QC, sample, shipment, and/or analytical problems encountered in processing the samples reported in the CSF.

2.4.5.1　The Contractor shall list the target analytes for the SDG.

2.4.5.2　The Contractor shall include any technical and administrative problems encountered, and the resolution or corrective actions taken. These problems may include, but are not limited to interference problems encountered during analysis, listing results from raw results less than the negative Contract Required Quantitation Limit (CRQL), and any problems with the analysis of samples.

2.4.5.3   Document the alternative temperature technique used, if applicable, to determine shipping container temperature if a temperature indicator bottle is not present in the shipping container.

2.4.5.4   The Contractor shall also provide equations for calibration curves with its fit expression (at least one equation or calibration curve per method), to allow the recalculation of sample results from raw instrument output.

2.4.5.5   The Contractor shall also include a discussion of any SOW Modified Analyses. This includes attaching a copy of the approved modification form to the SDG Narrative.

2.4.5.6   The Contractor shall also identify and explain any differences which exist between the Form(s) 1-IN and supporting documentation provided in the data package and those previously provided as Preliminary Results.

2.4.5.7   When submitting corrected data as "Additional Data" at the request of an EPA Regional data reviewer, the Contractor shall include a revised SDG Narrative documenting the reason(s) for the resubmittal.

2.4.5.8   The Contractor shall indicate if IEC Factors were applied during the ICP-AES analysis and if background corrections were applied, during the ICP-AES and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) analyses. If background corrections were applied, the Contractor shall indicate if raw data was generated prior to the application of the background corrections.

2.4.5.9   The Contractor shall document the use of collision or reaction cells for reducing ICP-MS interferences. The Contractor shall document: the type of cell and cell mode; the gas(es) used; any additional manufacturer-recommended setup or QC applied to establish analytical conditions (e.g., oxide ratios); list the analysis conditions applied to each analyte and internal standard (e.g., mass), along with any changes in the course of the analytical sequence; and any deliberate use of molecular species to avoid isobaric interferences (e.g., $^{75}As^{16}O$ at mass 91 to avoid $^{40}Ar^{35}Cl$ at mass 75).

2.4.5.10   The Contractor shall report the pH value for soil/sediment samples, if the measurement is requested.

2.4.6   SDG Cover Page

Cover Page for the inorganic analyses data shall include: Laboratory Name; Laboratory Code; Contract Number; Case Number; SDG Number; Modified Analysis Number (MA No.) (if appropriate); SOW Number; EPA Sample Numbers in alphanumeric order cross-referenced with Laboratory Sample ID numbers; and Analytical Method.

2.4.6.1   The SDG Cover Page shall contain the following statement, verbatim: "I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the SDG Narrative. Release of the data contained in this hardcopy Complete SDG File and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.4.7   ICP-AES Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for Toxicity Characteristic Leaching Procedure/Synthetic Precipitation Leaching Procedure (TCLP/SPLP), where applicable.

2.4.7.1   Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.7.2   Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.7.2.1   Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.7.2.2   Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.7.2.3   ICP Interference Check Sample [Form 4-IN]. Not required for Level 2a deliverables.

2.4.7.2.4   Matrix Spike Sample Recovery [Form 5A-IN]

2.4.7.2.5   Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

2.4.7.2.6   Duplicates [Form 6-IN]

2.4.7.2.7   Laboratory Control Sample [Form 7-IN]

2.4.7.2.8   ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

2.4.7.2.9   Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.7.2.10   ICP-AES Interelement Correction Factors [Form 10A-IN]. Not required for Level 2a deliverables.

2.4.7.2.11   ICP-AES Interelement Correction Factors [Form 10B-IN]. Not required for Level 2a deliverables.

2.4.7.2.12   Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.7.2.13   Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.7.2.14   Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.7.3   Raw Data – Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required Quality Assurance/Quality Control (QA/QC) measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF. The raw data for all samples shall include not only the results for the requested analyte(s), but also those for all the interferents [Exhibit D – Inductively Coupled Plasma – Atomic Emission Spectroscopy, Table 1 – Interferent and Analyte Concentrations Used for ICP-AES Interference Check Sample (ICS)].

The raw data shall also contain the results of any other element(s) which have been determined to interfere with the requested analytes(s).

2.4.7.3.1   Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. For example, if the instrument is applying an interelement correction for a reduced analyte list, the data used to calculate the correction must be present in the raw data. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.4.7.3.2   All raw data shall include concentration units.

2.4.7.3.3   Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.4.7.3.4   Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:

- Calibration standards;

- Initial and Continuing Calibration Blanks and Preparation Blanks;

- Initial and Continuing Calibration Verification standards, Interference Check Samples (ICSs), serial dilution samples, and Laboratory Control Samples (LCSs);

- Diluted and undiluted field samples;

- Duplicates;

- Spikes (matrix and post-digestion); and

- Instrument used.

2.4.7.4   Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for ICP-AES. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which QC samples (i.e., LCS, Preparation Blank) correspond to each batch digested; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

2.4.7.5   Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file identifiers (IDs), Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.

2.4.7.6   Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to the identity of: the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.

2.4.7.7   Extraction Logs for TCLP and SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction. For TCLP, include log for determination of extraction fluid, including sample weights and the initial and final pH determination.

2.4.7.8   Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for ICP-AES as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.

2.4.8   ICP-MS Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, and Analysis Logs, where applicable.

2.4.8.1   Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results of the requested analytes shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.8.2   Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.8.2.1   Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.8.2.2   Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.8.2.3   ICP Interference Check Sample [Form 4-IN]. Not required for Level 2a deliverables.

2.4.8.2.4   Matrix Spike Sample Recovery [Form 5A-IN]

2.4.8.2.5   Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

2.4.8.2.6   Duplicates [Form 6-IN]

2.4.8.2.7   Laboratory Control Sample [Form 7-IN]

2.4.8.2.8   ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

2.4.8.2.9   Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.8.2.10   ICP-MS Internal Standard Association [Form 11-IN]. Not required for Level 2a deliverables.

2.4.8.2.11   Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.8.2.12   ICP-MS Tune [Form 13-IN]. Not required for Level 2a deliverables.

2.4.8.2.13   ICP-MS Internal Standards Relative Intensity Summary [Form 14-IN]. Not required for Level 2a deliverables.

2.4.8.2.14   Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.8.2.15   Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.8.3   Raw Data – Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF. The raw data for all samples shall include not only the results for the requested analyte(s), but also those for all the interferents [Exhibit D – Inductively Coupled Plasma – Mass Spectrometry, Table 1 – Interferent and Analyte Concentrations Used for ICP-MS Interference Check Sample (ICS)].

The raw data shall also contain the results of any other element(s) or masses which have been determined to interfere with the requested analytes(s).

2.4.8.3.1   Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. For example, if the instrument is applying a correction for a reduced analyte list, the data used to calculate the correction must be present in the raw data. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.4.8.3.2   All raw data shall include concentration units.

2.4.8.3.3   Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.4.8.3.4   Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:

- Calibration standards;

- Initial and Continuing Calibration Blanks and Preparation Blanks;

- Initial and Continuing Calibration Verification standards, ICSs, serial dilution samples, and LCSs;

- Diluted and undiluted field samples;

- Duplicates;

- Spikes (matrix and post-digestion); and

- Instrument used.

2.4.8.4   Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for ICP-MS. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which QC samples (i.e., LCS, Preparation blank) correspond to each batch digested; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

2.4.8.5   Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file IDs, Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.

2.4.8.6   Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards and reagents. Standards shall be clearly labeled as to the identity of: the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.

2.4.8.7   Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for ICP-MS as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.

2.4.9   Mercury Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Digestion Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for TCLP/SPLP, where applicable.

2.4.9.1   Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results for mercury shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.9.2   Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.9.2.1   Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.9.2.2   Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.9.2.3   Matrix Spike Sample Recovery [Form 5A-IN]

2.4.9.2.4   Duplicates [Form 6-IN]

2.4.9.2.5   Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.9.2.6   Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.9.2.7   Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.9.2.8   Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.9.3   Raw Data – Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF.

2.4.9.3.1   Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.4.9.3.2   All raw data shall include absorbances or concentration units for mercury.

2.4.9.3.3   Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.4.9.3.4   Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 – Codes for Labeling Data, to unequivocally identify:

- Calibration standards;

- Initial and Continuing Calibration Blanks and Preparation Blanks;

- Initial and Continuing Calibration Verification standards;

- Diluted and undiluted field samples;

- Duplicates;

- Spikes (matrix); and

- Instrument used.

2.4.9.4   Digestion Logs (only required for the Level 3 deliverables). The digestion logs shall be submitted for each preparation procedure for mercury. These logs shall include: date; sample weights and volumes, with initial sample weight/volume clearly indicated; sufficient information to unequivocally identify which Calibration Standards or QC samples [e.g., Initial Calibration Verification (ICV), Preparation Blank] correspond to each batch digested; comments describing any significant sample changes or

reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH less than or equal to 2; PE preparation information (e.g., as-received PEs to final digestate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

2.4.9.5   Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; identification that includes electronic data file IDs, Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.

2.4.9.6   Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to: the identity of the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.

2.4.9.7   Extraction Logs for TCLP and SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction. For TCLP, include log for determination of extraction fluid, including sample weights and the initial and final pH determination.

2.4.9.8   Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for mercury as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.

2.4.10   Cyanide Sample Data Forms and Raw Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. All reporting forms shall be arranged in sequential order in increasing alphanumeric EPA Sample Number order, where applicable. The reporting forms shall be followed by the raw data, including sample, calibration, and QC data. This shall be followed by supporting documentation, including but not limited to: Distillation Logs, Standard and Reagent Preparation Logs, Analysis Logs, and Extraction Logs for SPLP, where applicable.

2.4.10.1   Inorganic Analysis Data Sheet [Form 1-IN]. Tabulated analytical results for cyanide shall be included. The validation and release of these results shall be authorized by a specific signed statement on the Cover Page. In the event that the Laboratory Manager cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.

2.4.10.2   Quality Control and Calibration Data

The QC summary for inorganic analysis shall contain the forms listed below. Please note some forms are not required for Level 2a deliverables.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

2.4.10.2.1   Initial and Continuing Calibration Verification [Form 2-IN]. Not required for Level 2a deliverables.

2.4.10.2.2   Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

2.4.10.2.3   Matrix Spike Sample Recovery [Form 5A-IN]

2.4.10.2.4   Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

2.4.10.2.5   Duplicates [Form 6-IN]

2.4.10.2.6   Method Detection Limit [Form 9-IN]. Not required for Level 2a deliverables.

2.4.10.2.7   Analysis Log [Form 12-IN]. Not required for Level 2a deliverables.

2.4.10.2.8   Initial Calibration [Form 15-IN]. Not required for Level 2a deliverables.

2.4.10.2.9   Initial Calibration Summary [Form 16-IN]. Not required for Level 2a deliverables.

2.4.10.3   Raw Data – Only required for Level 3 deliverables.

For each reported value, the Contractor shall include in the CSF all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the verification of method and instrument parameters submitted as a part of each CSF.

2.4.10.3.1   Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Bench Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the MDL. Raw data shall not be corrected for dilutions or volume adjustments. All instruments shall provide a legible hardcopy of the direct real-time instrument readout or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included.

2.4.10.3.2   All raw data shall include absorbances or concentration units for cyanide.

2.4.10.3.3   Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

2.4.10.3.4   Raw data shall be labeled with EPA Sample Numbers and appropriate codes, shown in Exhibit B, Table 5 - Codes for Labeling Data, to unequivocally identify:

- Calibration standards;

- Initial and Continuing Calibration Blanks and Preparation Blanks;

- Initial and Continuing Calibration Verification standards;

- Diluted and undiluted field samples;

- Duplicates;

- Spikes (matrix and post-distillation); and

- Instrument used.

2.4.10.4   Distillation Logs (only required for the Level 3 deliverables). The distillation logs shall be submitted as appropriate for each preparation procedure for cyanide. These logs shall include: date; sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; sufficient information to unequivocally identify which Calibration Standards and QC samples (e.g., ICV, Preparation Blank) correspond to each batch distilled; comments describing any significant sample changes or reactions which occurred during preparation shall be entered in the log and noted in the SDG Narrative; indication of pH greater than or equal to 12; PE preparation information (e.g., as-received PEs to final distillate); identification of the sample preparer(s) [signature(s)]; and sufficient information to identify the concentrations and volumes of reagents added to the samples.

2.4.10.5   Analysis Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic form shall be maintained for all analytical sequences to enable their reconstruction in time. The analysis logs shall record at a minimum: the date and time of analysis of each analysis within the sequence; electronic data file IDs; Lab Sample IDs or EPA Sample IDs; analyst identification; notation of QC failures and reasons; and sample dilutions.

2.4.10.6   Standard and Reagent Preparation Logs (only required for the Level 3 deliverables). Logbooks in hardcopy or electronic format shall be maintained for the preparation of all standards, reagents, and extraction fluids. Standards shall be clearly labeled as to: the identity of the analyte or analytes, the standard ID, concentration, date prepared, expiration date of the solution, special storage requirements if any, and the preparer's signature. Standards and reagents must be traceable. Dilutions from the primary standard and the calculations for determining their concentrations shall be recorded and verified by a second person.

2.4.10.7   Extraction Logs for SPLP (only required for Level 3 deliverables). Logbooks shall be submitted for any extraction performed by the Contractor. These shall include: the amount of aqueous and solid phases, percent solids determination, sample weight extracted, extraction fluid used, and start and end time of extraction.

2.4.10.8   Performance Evaluation (PE) Sample Instructions (only required for the Level 3 deliverable). If PE or PT audit samples are provided to the Contractor and analyzed for cyanide as part of the SDG, the Contractor shall submit a copy of the instructions that accompanied the sample(s) in the CSF.

2.5   Copy of Complete Sample Delivery Group File

        The laboratory shall provide a copy of the CSF and a PDF file to SMO,
        as specified in Table 1 – Deliverable Schedule, of this Exhibit.

2.6   Electronic Data Deliverables

        The Contractor shall provide the required electronic data deliverable
        as specified in Table 1 – Deliverable Schedule of this Exhibit.

2.6.1   Electronic Data Delivery in Staged Electronic Data Deliverable

        The Contractor shall provide an EDD in SEDD format for Levels 2a,
        2b, and 3. The EDD shall include analytical data for all samples in
        the SDG, as specified in Exhibit H – Format for Electronic Data
        Deliverables.

2.6.2   Portable Document Format of Complete Sample Delivery Group File

        The Contractor shall provide a complete copy of the CSF, and any
        additional or reconciled hardcopy deliverables, in a PDF file via
        EXES at https://epasmoweb.fedcsc.com, and follow the naming
        convention for the PDF file. The format of the PDF file should be
        HCD_Case Number_SDG Number_Contract Number_Submission Type.

2.6.2.1   The following identifiers are used based on submission type:

TABLE 2. PDF SUBMISSION IDENTIFIERS

Submission Type	Identifier
First Submission	FS
Replacement Submission (if a complete replacement of the first submission PDF is required)	RS
Reconciliation Submission	R# (The # character represents the number of the reconciliation. For example, the first reconciliation submission would be identified as R1.)
Additional Data Submission	A# (The # character represents the number of the additional data submissions. For example, the first additional data submission would be identified as A1.)

2.6.2.1.1   The PDF file shall be organized in accordance with the
             directions provided in Exhibit B, Section 2.0 of the SOW.

2.6.2.1.2   Inorganic data shall be bookmarked using a hierarchical
             bookmark structure (i.e., an overview or "parent" bookmark,
             and a subordinate or "child" bookmark nested underneath the
             "parent" bookmark). The required hierarchical structure is
             shown in Table 3 – Hierarchical Bookmark Structure.

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE

Group Bookmark	Parent Bookmark	Child Bookmark
SDG Cover Page, Sample TR/COC Records, Form DC-1, Form DC-2, and SDG Narrative		
ICP-AES Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		ICP Interference Check Sample
		Matrix Spike Sample Recovery
		Post-Digestion Spike Sample Recovery
		Duplicates
		Laboratory Control Sample
		ICP-AES Serial Dilutions
		Method Detection Limits
		ICP-AES Interelement Correction Factors
		Analysis Log
		Initial Calibration
		Initial Calibration Summary
	Raw Data	ICP-AES Raw Data
		ICP-AES Digestion Logs
		Preparation and Analysis Logbooks
		TCLP/SPLP Logbooks
		PE/PT Instruction Forms
ICP-MS Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		ICP Interference Check Sample
		Matrix Spike Sample Recovery
		Post-Digestion Spike Sample Recovery
		Duplicates
		Laboratory Control Sample
		ICP-MS Serial Dilutions
		Method Detection Limits
		ICP-MS Internal Standard Association
		Analysis Log
		ICP-MS Tune
		ICP-MS Internal Standard Relative Intensity Summary
		Initial Calibration
		Initial Calibration Summary

TABLE 3. HIERARCHICAL BOOKMARK STRUCTURE (CON'T)

Group Bookmark	Parent Bookmark	Child Bookmark
ICP-MS Data (Cont'd)	Raw Data	ICP-MS Raw Data
		ICP-MS Digestion Logs
		Preparation and Analysis Logbooks
		PE/PT Instruction Forms
Mercury Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		Matrix Spike Sample Recovery
		Duplicates
		Method Detection Limits
		Analysis Log
		Initial Calibration
		Initial Calibration Summary
	Raw Data	Mercury Raw Data
		Mercury Digestion Logs
		Preparation and Analysis Logbooks
		TCLP/SPLP Logbooks
		PE/PT Instruction Forms
Cyanide Data	Sample Data	Inorganic Analysis Data Sheet in increasing alphanumeric EPA Sample Number order
	QC Summary	Initial and Continuing Calibration Verification
		Blanks
		Matrix Spike Sample Recovery
		Post-Distillation Spike Sample Recovery
		Duplicates
		Method Detection Limits
		Analysis Log
		Initial Calibration
		Initial Calibration Summary
	Raw Data	Cyanide Raw Data
		Cyanide Distillation Logs
		Preparation and Analysis Logbooks
		SPLP Logbooks
		PE/PT Instruction Forms
Receiving Documents, Transfer Records, and Miscellaneous	Additional Documents	Percent Solids Log
		Receiving Logbooks
		Internal Sample, Digestate, and Distillate Transfer Chain-of-Custody Records
		Communication Logs

2.7   Preliminary Results

The Form(s) 1-IN data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form(s) 1-IN as "Preliminary Results" under the form title (i.e., under Inorganic Analysis Data Sheet). The Contractor shall also include a disclaimer on all Form(s) 1-IN stating that the "Data results contained on this Form 1-IN are for screening purposes only, and may not have been validated for CLP criteria." Sample TR/COC Records and SDG Cover Page (per Exhibit B Section 2.7.1) shall be submitted with the Preliminary Results.

2.7.1   The Contractor shall submit the SDG Cover Page following the specifications in Exhibit B, Sections 2.4.6 and 3.4.1. The SDG Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The SDG Cover Page shall contain the following statement, verbatim: "I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the SDG Narrative. Release of the data contained in this hardcopy Data Package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

2.8   Method Detection Limits and Interelement Correction Factors

The Contractor shall perform and report determination of the MDLs by the method specified in Exhibit D - Analytical Methods for each instrument used under this contract. Results for the verification of method parameters for the current period shall be submitted using Form 9-IN.

The Contractor shall also perform and report ICP-AES IEC factors (including method of determination) and wavelengths used. Results for the verification of method parameters for the current period shall be submitted using Forms 10A-IN and 10B-IN.

The Contractor shall deliver all determined MDLs to SMO and QATS electronically in the format described in Appendix A – Format Characteristics for Method Detection Limit Study Data, of Exhibit H – Format for Electronic Data Deliverables, according to the delivery schedule specified in Table 1 – Deliverable Schedule, of Exhibit B – Reporting and Deliverables Requirements.

Submission of the study data for the determination of method and instrument parameters, to QATS only, shall include the data used to determine the values reported as well as, for the IECs, the standard preparation logs, sample preparation logs, and analysis logs with analytical sequences. The Contractor shall provide MDL and IEC raw data including sample, calibration, and QC data and supporting documentation, including, but not limited to: Digestion/Distillation Logs, Standard and Reagent Preparation Logs, and Analysis Logs, where applicable, to QATS only, according to the delivery schedule specified in Table 1 – Deliverable Schedule, of Exhibit B – Reporting and Deliverables Requirements.

3.0   FORM INSTRUCTIONS

3.1   Introduction

        This section contains specific instructions for the completion of all
        required Inorganic Data Reporting Forms.

3.2   General Information

        Values shall be reported on the hardcopy forms according to the
        respective form instructions in this section.

3.2.1   The data reporting forms discussed in Exhibit B, Section 3.4, and
        presented in Exhibit B, Section 4.0, have been designed in
        conjunction with the electronic data format specified in Exhibit H –
        Format for Electronic Data Deliverables. Information entered on
        these forms shall **not** exceed the size of the field given on the
        form, including such laboratory-generated items as "Lab Name" and
        "Lab Sample ID". See Table 4 - Required Forms for Reporting Level,
        for a listing of required forms by reporting level.

                   TABLE 4. REQUIRED FORMS FOR REPORTING LEVEL

Level	Required Forms
SEDD 2a	Forms 1, 3, 5, 6, 7, 8
SEDD 2b	Forms 1-16 (all Forms)
SEDD 3	Forms 1-16 (all Forms)

3.2.2   All characters which appear on the data reporting forms presented in
        Section 4.0 shall be reproduced by the Contractor when submitting
        data, and the format of the forms submitted shall provide exactly
        the same information as that shown in the contract. No information
        may be added, deleted, or moved from its specified position. The
        names of various fields and analytes (i.e., "Lab Code", "Preparation
        Batch") shall appear as they are listed in Exhibit B – Reporting and
        Deliverables Requirements, and Exhibit C – Inorganic Target Analyte
        List and Contract Required Quantitation Limits, of this SOW.

3.2.3   Rounding Rules

        For rounding off numbers to the appropriate level of precision,
        observe the following common rules. If the figure following those
        to be retained is greater than or equal to 5, the absolute value of
        the result is to be rounded up; otherwise the absolute value of the
        result is rounded down. For example, -0.4365 rounds to -0.44 and
        –2.3464 rounds to -2.3. Also see "Rounding Rules" in Exhibit G –
        Glossary of Terms.

3.2.3.1 Before evaluating a number for being in control or out of control
        of a certain limit (other than the CRQL), the number evaluated
        shall be rounded using the above rounding rules to the
        significance reported for that limit. For example, the control
        limit for an ICV is plus or minus 10% of the true value. Then a
        calculated percent recovery (%R) of 110.46 shall be reported on
        Form 2-IN as 110, which is within the control limits of 90-110.
        On the other hand, a calculated %R of 110.50 shall be reported on
        Form 2-IN as 111, which is not within the 90-110 percent control
        limits.

3.2.4   Significant Figures

All results shall be transcribed to Inorganic Forms 1-IN through 16-IN from the instrument raw data to two significant figures if the value is less than 10, or three significant figures if the value is greater than or equal to 10 as described in Exhibit B – Reporting and Deliverables Requirements, and Exhibit H – Format for Electronic Data Deliverables. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. The instrument raw data files contain the raw data values. The hardcopy raw data may be a rounded or truncated representation of the instrument raw data.

3.3   Header and General Form Information

Six pieces of information are common to the header section of each data reporting form. These are Lab Name, Contract, Lab Code, Case Number (Case No.), Modified Analysis Number (MA No.), and SDG Number (SDG No.). Except as noted below for MA No., this information shall be entered on every form and shall match on all forms.

3.3.1   "Lab Name" shall be the name chosen by the Contractor to identify the laboratory.

3.3.2   "Contract" is the number of the EPA contract under which the analyses were performed.

3.3.3   "Lab Code" is an alphanumeric abbreviation, assigned by the EPA, to identify the laboratory and aid in data processing. This Lab Code will be assigned by the EPA at the time a contract is awarded and shall not be modified by the Contractor, except at the direction of the EPA Contracting Officer (CO). If a change of name or ownership occurs at the laboratory, the Lab Code will remain the same unless and until the Contractor is directed by the EPA CO to use another EPA-assigned Lab Code.

3.3.4   "Case No." is the SMO-assigned Case Number associated with the sample, and reported on the TR/COC Record or sample shipping paperwork.

3.3.5   "MA No." is the EPA-assigned number for analyses performed for an analytical method under the Modified Analysis clause in Exhibit A – Summary of Requirements. If samples are to be analyzed under the Modified Analysis clause, the Contractor shall list the modification reference number on all forms. If the analyses have no modified requirements, leave the "MA No." field blank.

3.3.6   "SDG No." is the SDG Number.

3.3.7   "EPA SAMPLE NO." appears either in the header information of the form or as the left column of a table summarizing data from a number of samples.

3.3.7.1   All samples, leachates, blanks, matrix spikes, post-digestion/ distillation spikes, duplicates, and serial dilutions shall be identified with an EPA Sample Number. For samples, an EPA Sample Number is the unique identifying number given on the TR/COC Record or sample shipping records that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 5 - Codes for Labeling Data, must be used.

TABLE 5. CODES FOR LABELING DATA[1,2,3,4]

Sample	Sample Number
Sample in SDG (TCLP/SPLP Leachate included)	XXXXXX
Sample or Laboratory QC Not Part of the SDG	ZZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Post-Digestion/Distillation Spike	XXXXXXA
Instrument Calibration Standards	S##
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV###
Continuing Calibration Blank	CCB###
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
Laboratory Control Sample	LCS###
Preparation Blank (Aqueous/Water)	PBW###
Preparation Blank (Soil/Sediment)	PBS###
Preparation Blank (Wipe)	PBF###
Leachate Extraction Blank	LEB###
ICP-MS Tune Check	TUNE

Footnotes:

[1] Instrument QC samples must not be reported as ZZZZZZ.

[2] For samples received under the CLP for inorganic analyses, the sample number will begin with an "M".

[3] The suffix that follows the "S" for the standards indicates the sequence number of the standard analysis. Beginning with S01 and continuing to the last standard analyzed.

[4] Within an analytical method, the three-character suffix (###) shall be unique for each instance of each sample type within an SDG. The Contractor may achieve this by replacing the suffix with one to three alpha-numeric characters.

3.3.7.2   These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MA1111 is the lowest (considering both alpha and numeric characters) EPA Sample Number within the SDG, it would be entered in the first EPA Sample Number field. Samples would be listed below it, in ascending sequence - MA1111, MA1111D, MAB124, MAB125, MAC111, etc.

3.3.8   "Matrix" is the matrix of the sample. Enter "Soil" for soil/sediment samples, "Water" for aqueous/water and leachate samples, and "Wipe" for wipes, as appropriate.

3.3.9   "Analytical Method" is the method used to analyze the sample. Enter "ICP-AES", "ICP-MS", "CVAA", or "Spectrophotometry", as appropriate.

3.3.10   "Run Batch" is the unique identifier of the analytical sequence from the EDD. Report the RunBatch identifier for the Analytical Sequence reported on the form.

3.3.11   "Preparation Batch" is the unique identifier of the preparation batch from the EDD. Report the PreparationBatch identifier for the preparation reported on the form.

3.3.12   "Preparation Method" is the method used to prepare the samples for analysis. Report the preparation method reported on the form as specified below:

> 200.7: ICP-AES aqueous/water samples
> 200.8: ICP-MS aqueous/water samples and soil/sediment samples
> 3050B: ICP-AES soil/sediment samples and ICP-AES wipe samples
> 7470A: Mercury aqueous/water samples
> 7471B: Mercury soil/sediment samples
> Midi-distillation_Aqueous: Cyanide aqueous/water samples
> Midi-distillation_Soil: Cyanide soil/sediment samples
> Micro-distillation_Aqueous: Cyanide aqueous/water samples
> Micro-distillation_Soil: Cyanide soil/sediment samples

3.3.13   "Concentration Units" are the units in which the analytical result is reported. Enter "µg/L", "mg/L", "mg/kg", or "µg" as appropriate.

3.3.14   "%Solids" is the percent solids of the soil/sediment sample as determined by the procedure in Exhibit D – General Inorganic Analysis.

3.3.15   "Instrument ID" is the unique identifier of the instrument with which analysis is performed.

3.3.16   "Analyte" is identified in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits, and must be reported in the order given in Exhibit C.

3.4   Reporting Forms

3.4.1   SDG Cover Page

3.4.1.1   Purpose

This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager or designee to authorize and release all data and deliverables associated with the SDG.

3.4.1.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.1.2.1   For samples analyzed using this SOW, enter "ISM02.4" for the SOW Number.

3.4.1.2.2   Under column "EPA Sample No.", enter each EPA Sample Number.

3.4.1.2.3   Under column "Lab Sample ID", enter each Laboratory sample identifier.

3.4.1.2.4   Under column "Analysis Method", enter an "X" under each Analytical Method scheduled for analysis for each EPA Sample Number.

3.4.1.2.5   Each SDG Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager's designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.

3.4.2   Inorganic Analysis Data Sheet [Form 1-IN]

3.4.2.1   Purpose

This form is used to tabulate and report sample analysis results for inorganic target analytes per analytical method (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits).

3.4.2.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.2.2.1   "Lab Sample ID", enter the Laboratory sample identifier.

3.4.2.2.2   "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the TR/COC Record (i.e., the VTSR).

3.4.2.2.3   Under column "CAS No.", enter the Chemical Abstracts Service (CAS) Number for each analyte as listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

3.4.2.2.4   Under column "Concentration", enter for each analyte, the value of the result if the concentration or mass is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions. If the concentration is less than the adjusted MDL enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions.

3.4.2.2.5   Under column "Q", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included in the SDG Narrative.

3.4.2.2.5.1   The MDL obtained for a given preparation method, analysis method, and instrument shall be used for the qualification of the results for samples associated with that preparation method, analysis method, and instrument. Serial dilution and post-digestion/distillation spike results shall be qualified using the MDL and CRQL values utilized for the corresponding field sample.

All three values (i.e., the instrument reading, CRQL, and MDL) shall be converted to the same units prior to determining the appropriate qualifier.

3.4.2.2.5.2   Specified entries and their meanings are as follows:

X: The reported value is estimated due to interferences.

*: QC analyses are outside control limits.

D: The reported value is from a dilution.

J: The reported value was less than the CRQL, but greater than or equal to the MDL.

U: The result was less than the MDL. For Hardness, if the results for both Ca and Mg were less than their respective MDLs.

3.4.2.2.6   Under column "Date Analyzed", for each analyte reported, enter the date of the analysis the result is being reported from as MM/DD/YYYY.

3.4.2.2.7   Under column "Time Analyzed", for each analyte reported, enter the time of the analysis the result is being reported from in military time (HHMM).

3.4.2.2.8   In the "Comments" field, note any significant changes that occur during sample preparation (e.g., emulsion formation), any sample-specific comments concerning the analyte results, and any raw instrument results that are less than the negative CRQL (-CRQL). These notes shall also be included the SDG Narrative.

3.4.3   Initial and Continuing Calibration Verification [Form 2-IN]. This form is not required for Level 2a deliverables.

3.4.3.1   Purpose

This form is used to report analyte recoveries from calibration verification solutions.

3.4.3.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.3.2.1   "Initial Calibration Verification Source" and the "Continuing Calibration Verification Source" identify the manufacturer and the solution (lot) used.

Use additional Form(s) 2-IN if more calibration verification sources were used.

3.4.3.2.2   Under column "Initial Calibration Verification", enter the following:

3.4.3.2.2.1   "ID", enter the EPA Sample Number of the ICV reported on the form.

3.4.3.2.2.2   Under column "True", enter the expected concentration or true amount of each analyte in the ICV Solution.

3.4.3.2.2.3   Under column "Found", enter the concentration of each analyte measured in the ICV Solution.

3.4.3.2.2.4   Under column "%R", enter the percent Recovery (%R) (to the nearest whole number) calculated using the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{Found(ICV)}{True(ICV)} \times 100$$

WHERE,
Found(ICV)   = The found concentration of the analyte in the ICV Solution
True (ICV)   = The true amount of the analyte in the ICV Solution

3.4.3.2.2.5 Under column "%RSD", enter the percent Relative Standard Deviation (%RSD) (to the nearest whole number) of the replicates for ICP-AES and ICP-MS analysis. Leave this column blank for Hg and CN analysis.

3.4.3.2.2.5.1 Calculate the %RSD from all replicate integrations using the following equation:

EQ. 2 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

WHERE,
SD = Standard deviation of ICV replicates (per analyte) from EQ. 3

$\overline{X}$ = Mean value of the ICV replicates (per analyte) from EQ. 4

3.4.3.2.2.5.2 Equation 3 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 3 Standard Deviation Calculation

$$SD = \sqrt{\frac{\sum\limits_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

WHERE,
X_i = Each individual value used to calculate the mean

$\overline{X}$ = The mean of n values from EQ. 4

n = Total number of values

3.4.3.2.2.5.3 Equation 4 is the general formula for the mean of a set of values ($\overline{X}$).

EQ. 4 Mean Value Calculation

$$\overline{X} = \frac{\sum\limits_{i=1}^{n} X_i}{n}$$

WHERE,
X_i = Each individual value used to calculate the mean

n = Total number of values

3.4.3.2.3 Under column "Continuing Calibration Verification", enter the following:

3.4.3.2.3.1 "ID", enter the EPA Sample Numbers of the CCVs reported on the form.

3.4.3.2.3.2 Under column "True", enter the expected concentration or true amount of each analyte in the CCV Solution.

3.4.3.2.3.3 Under column "Found", enter the concentration of each analyte measured in the CCV Solution.

3.4.3.2.3.4   Under column "%R", enter the percent recovery (to the nearest whole number) calculated using the following equation:

EQ. 5 CCV Percent Recovery

$$\%R = \frac{Found(CCV)}{True(CCV)} \times 100$$

WHERE,
Found(CCV)   = The found concentration of the analyte in the CCV Solution
True(CCV)   = The true amount of the analyte in the CCV Solution

3.4.3.2.3.5   Under column "%RSD", enter the %RSD (to the nearest whole number) of the replicates for each CCV for ICP-AES and ICP-MS analysis. Calculate the value using Equation 2 with the CCV replicates. Leave these columns blank for Hg and CN analysis.

3.4.3.2.4   The order of reporting ICVs and CCVs for each analyte shall follow the chronological order in which the standards were analyzed. Start with the first Form 2-IN and report from the left to the right, continuing to the following Form(s) 2-IN as appropriate. For example, the first ICV shall be reported on the first Form 2-IN.

In an analytical sequence where three CCVs were analyzed, the first CCV shall be reported in the left CCV column on the first Form 2-IN and the second CCV shall be reported in the right column of the same form. The third CCV shall be reported in the left CCV column of the second Form 2-IN. On the second Form 2-IN, the ICV column and the right CCV column shall be left empty in this example. In the previous example, if a second analytical sequence for an analyte was needed, the ICV of that analytical sequence shall be reported on a third Form 2-IN and the CCVs follow in the same fashion as explained before.

NOTE: In the case where two wavelengths are used for an analyte, all ICV and CCV results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.

3.4.4   Blanks [Form 3-IN]. For Level 2a deliverables, only Preparation Blank data is required.

3.4.4.1   Purpose

This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), Preparation Blank, and Leachate Extraction Blank (LEB).

3.4.4.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.4.2.1   "Preparation Blank Matrix", enter appropriate matrix (water, soil, or wipe). No abbreviations or other matrix descriptors may be used.

3.4.4.2.2   "Preparation Blank Concentration Units", enter appropriate concentration units (µg/L for water, mg/L for TCLP leachates, mg/kg for soil, or µg for wipes).

3.4.4.2.3   Under column "Initial Calibration Blank", enter the following:

3.4.4.2.3.1   "ID", enter the EPA Sample Number of the ICB reported on the form.

3.4.4.2.3.2   Under "Initial Calibration Blank", enter the concentration of each analyte in the most recent ICB.

3.4.4.2.3.2.1   Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.

3.4.4.2.3.2.2   Under column "Q", enter "J" if the absolute value of the analyte concentration is less than the CRQL for aqueous/water but greater than or equal to the MDL (in µg/L) determined for the default aqueous/water preparation method on that particular instrument.

     For prepared calibration blanks (e.g., mercury and cyanide), the CRQL for aqueous/water, and the MDL (in µg/L or converted to µg/L) for the preparation method, analysis, and instrument shall be used.

     Enter "U" if the absolute value of the analyte in the blank is less than the MDL (in µg/L or converted to µg/L) obtained from the default aqueous/water preparation method on that instrument (unprepared blanks) or determined for the preparation method (prepared blanks).

3.4.4.2.4   Under column "Continuing Calibration Blank", enter the following:

3.4.4.2.4.1   "ID", enter the EPA Sample Numbers of the CCBs reported on the form.

3.4.4.2.4.2   Under "Continuing Calibration Blank", enter the concentration of each analyte detected in the first required CCB analyzed after the ICB.

3.4.4.2.4.2.1   Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.

3.4.4.2.4.2.2   Under column "Q", enter any appropriate qualifier, as explained in Section 3.4.4.2.3.2.2.

3.4.4.2.5   Under column "Preparation Blank/Leachate Extraction Blank", enter the following:

3.4.4.2.5.1   "ID", enter the EPA Sample Number of the Preparation Blank or LEB reported on the form.

3.4.4.2.5.2   Under "Preparation Blank/Leachate Extraction Blank", enter the concentration of each analyte in the Preparation Blank or LEB.

3.4.4.2.5.2.1   Enter the concentration or mass (positive or negative) for each analyte, if the absolute value of the concentration or mass is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration or mass is less than the appropriate MDL.

3.4.4.2.5.2.2   Under the column "Q", enter the appropriate qualifier, as explained in Section 3.4.4.2.3.2.2.

3.4.4.2.6   The order of reporting ICBs and CCBs for each analyte shall follow the chronological order in which the blanks were analyzed, starting with the first Form 3-IN and reporting from left to right and continuing to additional Form(s) 3-IN. If LEBs are analyzed, they shall be reported on a separate Form 3-IN from any Preparation Blanks.

            NOTE: In the case where two wavelengths are used for an analyte, all ICB, CCB, and Preparation Blank and LEB results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.

3.4.5   ICP Interference Check Sample [Form 4-IN]. This form is not required for Level 2a deliverables.

3.4.5.1   Purpose

        This form is used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.

3.4.5.2   Instructions

        Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.5.2.1   "ICSA Source" and "ICSB Source", identify the manufacturer and the solution (lot) used. For EPA solutions, include the source name and number (e.g., ICSA-1206) as provided in the accompanying solution instructions.

3.4.5.2.2   Under column "True ICSA", enter the expected concentration or true amount of each analyte present in ICSA. Enter "0" for each analyte with no specified true value in ICSA.

3.4.5.2.3   Under column "True ICSAB", enter the expected concentration or true amount of each analyte present in ICSAB. Enter "0" for each analyte with no specified true value in ICSAB.

3.4.5.2.4   Under column "Found ICSA", enter the measured concentration (positive, negative, or zero) for each analyte and interferent. Enter the concentration of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of ICSA as required in Exhibit D. For ICP-MS, do not enter the interferent elements carbon, chloride, molybdenum, phosphorus, sulfur, and titanium. Report as provided in the instructions accompanying the material.

3.4.5.2.5   Under column "Found ICSA %R", enter the value of the percent recovery (to the nearest whole number) calculated for True ICSA greater than zero using the following equation. If "True ICSA" equals zero, leave field blank.

EQ. 6 ICSA Percent Recovery

$$\%R = \frac{Found(ICSA)}{True(ICSA)} \times 100$$

WHERE,

Found(ICSA) = The found concentration of the analyte in the ICSA Solution

True(ICSA) = The true amount of the analyte in the ICSA Solution

3.4.5.2.6 Under column "Found ICSAB", enter the measured concentration (positive, negative, or zero) for each analyte and interferent. Enter the concentration of each analyte and interferent for ICP-AES, and of each analyte and interferent for ICP-MS in the initial analysis of ICSB as required in Exhibit D. For ICP-MS, do not enter the interferent elements carbon, chloride, molybdenum, phosphorus, sulfur, and titanium. Report as provided in the instructions accompanying the material.

3.4.5.2.7 Under column "Found ICSAB %R", enter the percent recovery (to the nearest whole number) calculated for True ICSAB greater than zero using the following equation. If "True ICSAB" equals zero, leave field blank.

EQ. 7 ICSAB Percent Recovery

$$\%R = \frac{Found(ICSAB)}{True(ICSAB)} \times 100$$

WHERE,

Found (ICSAB) = The found concentration of the analyte in the ICSAB Solution

True (ICSAB) = The true amount of the analyte in the ICSAB Solution

3.4.5.2.8 If more ICS analyses were required, submit additional Form(s) 4-IN as appropriate.

3.4.5.2.8.1 The order of reporting ICSs for each analyte shall follow the chronological order in which the standards were analyzed, starting with the first Form 4-IN and continuing to the following Form(s) 4-IN as appropriate.

NOTE: In the case where two wavelengths are used for an analyte, all ICSA and ICSAB results of one wavelength from all analyses shall be reported before proceeding to report the results of the second wavelength used.

3.4.6 Matrix Spike Sample Recovery [Form 5A-IN]

3.4.6.1 Purpose

This form is used to report results for the pre-digestion/ distillation spike.

3.4.6.2 Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.6.2.1   Under column "Control Limit %R", enter "75-125" if the sample result is less than or equal to four times the Spike Added (SA) value. If the sample result is greater than four times the SA value, leave this field empty.

3.4.6.2.2   Under column "Spiked Sample Result (SSR)", enter the measured value, in appropriate units, for each relevant analyte in the matrix spike sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.

3.4.6.2.3   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.6.2.4   Under column "Sample Result (SR)", enter the measured value for each required analyte in the sample (reported in "EPA SAMPLE NO." box) on which the matrix spike was performed. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.

3.4.6.2.5   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.6.2.6   Under column "Spike Added (SA)", enter the expected concentration or true amount of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "SA" concentration is specified in the contract, then the value added and reported shall be the specific concentration, corrected for spiked sample weight and percent solids or spiked sample volume.

3.4.6.2.7   Under column "%R", enter the percent recovery (to the nearest whole number) for all spiked analytes calculated using the following equation. The percent recovery shall be reported, whether it is negative, positive, or zero.

                EQ. 8 Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

        WHERE,

SSR   =   Spiked Sample Result (µg/L or mg/kg)
SR   =   Sample Result (original) (µg/L or mg/kg). When the sample concentration is less than the MDL, use SR=0.
SA   =   Spike Added Theoretical Result (µg/L or mg/kg)

3.4.6.2.8   Under column "Q", enter "*" if the Spike Percent Recovery (%R) is out of the control limits (75-125%) and the Sample Result (SR) is less than or equal to four times the SA.

3.4.6.2.9   If different samples were used for spike sample analysis of different analytes, additional Form(s) 5A-IN shall be submitted for each sample as appropriate.

3.4.6.2.9.1   In the instance where there is more than one spike sample per matrix, per SDG, if one spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

3.4.7   Post-Digestion/Distillation Spike Sample Recovery [Form 5B-IN]

3.4.7.1   Purpose

This form is used to report results for the post-digestion/distillation spike recovery which is based upon the addition of a known quantity of analyte to an aliquot of the digested or distilled sample.

3.4.7.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.7.2.1   Under column "Control Limit %R", enter "75-125" if a Post-Digestion Spike was required for the analyte.

3.4.7.2.2   Under column "Spiked Sample Result (SSR)", enter the measured value for each analyte in the post-digestion/distillation spike sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.

3.4.7.2.3   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.7.2.4   Under column "Sample Result (SR)", enter the measured value for the concentration of each analyte in the sample (reported in "EPA SAMPLE NO." box) on which the spike was performed. Enter the value if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the adjusted MDL.

3.4.7.2.5   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.7.2.6   Under column "Spike Added (SA)", enter the expected concentration or true amount of each analyte added to the sample. If the "SA" concentration is specified in the contract, the value added and reported shall be that specific concentration.

3.4.7.2.7   Under column "%R", enter the percent recovery (to the nearest whole number) for all spiked analytes using the following equation. Percent recovery shall be reported, whether it is negative, positive, or zero.

EQ. 9 Post-Digestion/Distillation Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,
SSR = Spiked Sample Result (µg/L or mg/kg)
SR   = Sample Result (original) (µg/L or mg/kg). When the sample concentration is less than the MDL, use SR=0.
SA   = Spike Added Theoretical Result (µg/L or mg/kg)

3.4.7.2.8   If different samples were used for spike sample analysis of different analytes, additional Form(s) 5B-IN shall be submitted for each sample as appropriate.

3.4.7.2.9   Under column "Q", enter "*" if the Spike %R is out of the control limits (75–125%).

3.4.8   Duplicates [Form 6-IN]

3.4.8.1   Purpose

The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for all analyte results.

3.4.8.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.8.2.1   Under column "Control Limit", enter the CRQL (corrected for the original sample weight and percent solids, if necessary) for the analyte if either the sample or duplicate value was less than 5 times the CRQL. If the sample and duplicate values were greater than or equal to 5 times the CRQL, or if the sample and duplicate values were less than the CRQL, leave the field empty.

3.4.8.2.2   Under column "Sample (S)", enter the measured value for the concentration of each analyte in the sample (reported in "EPA SAMPLE NO." box) on which a duplicate analysis was performed. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.

3.4.8.2.3   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.8.2.4   Under column "Duplicate (D)", enter the measured value for each analyte in the duplicate sample. Enter the value of the result if the concentration is greater than or equal to the MDL adjusted if necessary and corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the adjusted MDL.

3.4.8.2.5   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.8.2.6   For soil/sediment samples, the concentration of the original sample shall be computed using the weight and percent solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample, but the percent solids of the original sample.

3.4.8.2.7   Under column "RPD", enter the absolute value (to the nearest whole number) of the Relative Percent Difference (RPD) for all analytes detected above the CRQL in either the sample or the duplicate, calculated using the following equation:

EQ. 10 Duplicate Sample Relative Percent Difference

$$RPD = \frac{\left| S - D \right|}{(S + D)/2} \times 100$$

WHERE,
S   = Sample result
D   = Duplicate result

If the analyte concentration is less than the MDL in either "S" or "D", a value of zero shall be substituted for "S" or "D". If the analyte concentration is less than the CRQL in both "S" and "D", leave the "RPD" field empty.

3.4.8.2.8   Under column "Q", enter "*" if the duplicate analysis for the analyte is outside control limits. If both sample and duplicate values are greater than or equal to 5 times the CRQL, then the RPD must be less than or equal to 20% to be in control. If either the sample or duplicate value is less than 5 times the CRQL, then the absolute difference between the sample and duplicate values shall be less than the CRQL to be in control. If both values are below the CRQL, then no control limit is applicable.

3.4.9   Laboratory Control Sample [Form 7-IN]

3.4.9.1   Purpose

This form is used to report results for the aqueous/water, soil/sediment, and wipe LCSs.

3.4.9.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.9.2.1   Under column "True", enter the value of the expected concentration or true amount of each analyte in the LCS.

3.4.9.2.2   Under column "Found", enter the concentration of each analyte found in the LCS.

3.4.9.2.3   Under column "%R", enter the percent recovery (to the nearest whole number) calculated using the following equation:

EQ. 11 LCS Percent Recovery

$$\%R = \frac{Found\ (LCS)}{True\ (LCS)} \times 100$$

WHERE,
Found (LCS) = The found concentration at each analyte in the LCS. If the analyte concentration is less than the MDL, a value of zero shall be substituted for the Found LCS.
True (LCS)   = The true amount of each analyte in the LCS

3.4.9.2.4   Submit additional Form(s) 7-IN as appropriate if more than one LCS was required.

3.4.10   ICP-AES and ICP-MS Serial Dilutions [Form 8-IN]

3.4.10.1   Purpose

This form is used to report results for ICP-AES and ICP-MS serial dilutions.

3.4.10.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.10.2.1   Under column "Initial Sample Result (I)", enter the measured value, corrected for any dilutions, for each analyte. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.

3.4.10.2.2   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.10.2.3   Under column "Serial Dilution Result (S)", enter the measured value for each analyte in the diluted sample. Enter the value if the concentration is greater than or equal to the adjusted MDL; or enter the adjusted CRQL if the concentration is less than the adjusted MDL.

3.4.10.2.4   Under column "Q", enter the result qualifier as identified in Section 3.4.2.2.5.

3.4.10.2.5   Under column "% Difference", enter the absolute value (to the nearest whole number) of the percent difference, between the original sample and the diluted sample (adjusted for dilution) using the following equation:

EQ. 12 Serial Dilution Percent Difference

$$\% \text{ Difference} = \frac{|\text{ I } - \text{ S }|}{\text{I}} \times 100$$

WHERE,
I   =   Initial sample result. If the analyte concentration is less than the MDL concentration, leave the "% Difference" field empty.
S   =   Serial dilution result. If the analyte concentration is less than the MDL, a value of zero shall be substituted for "S".

3.4.10.2.6   Under column "Q", enter "*" if the percent difference is greater than 10% and the original sample concentration (reported on Form 1-IN) is greater than 50 times the adjusted MDL.

3.4.11   Method Detection Limit [Form 9-IN]

3.4.11.1   Purpose

This form documents the MDL for each preparation method and instrument that the Contractor used to obtain data for the SDG. Only the methods, instruments, and wavelengths used to generate data for the SDG shall be included. A copy of the MDLs reported on Form(s) 9-IN shall be included with each CSF.

3.4.11.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.11.2.1   Under column "Wavelength/Mass", enter the wavelength in nanometers (nm) or the mass in atomic mass units (u) for each analyte for which an MDL has been established. If more than one wavelength or mass is used for an analyte, use additional Form(s) 9-IN as appropriate to report the MDLs.

3.4.11.2.2   Under column "MDL", enter the MDL as determined by the Contractor for each analyte analyzed by the instrument for which the ID is listed on this form.

When calculating MDL values, always round up to the appropriate significant figure (e.g., 14.81 rounds to 14.9 and 146.6 rounds to 147). This deviation from the rounding rule is necessary to prevent the reporting of detected values for results that fall in the noise region of the calibration curve.

NOTE: Zeroes used to set the decimal point in a number less than one are not significant, but all trailing zeroes are significant.

For example, a calculated MDL value of 0.074 µg/L will be reported as 0.074 and a calculated MDL value of 0.1 or 0.08 will be reported as 0.10 and 0.080, respectively.

3.4.11.2.3   The MDLs for Hardness, TCLP, or SPLP are not required to be reported.

3.4.11.2.4   Under column "Date Analyzed", enter the date analyzed (formatted as MM/DD/YYYY) for the analyte. Note that the date shall not exceed the analysis dates in the CSF or precede them by more than one year.

3.4.12   ICP-AES Interelement Correction Factors [Form 10A-IN]. This form is not required for Level 2a deliverables.

3.4.12.1   Purpose

This form documents for each ICP-AES instrument the IEC factors applied by the Contractor to obtain data for the SDG. A copy of the results of the IEC factors shall be included with each CSF on Form 10A-IN and Form 10B-IN as appropriate.

3.4.12.2   Instructions

Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.12.2.1   "Date", enter the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use.

3.4.12.2.2   Under column "Wavelength", enter the wavelength in nm used for each ICP-AES analyte. If more than one wavelength is used, submit additional Form(s) 10A-IN or Form(s) 10B-IN as appropriate.

3.4.12.2.3   Under columns "Al", "Ca", "Fe", and "Mg", enter the correction factor (negative, positive, or zero) for each ICP-AES analyte to the number of decimal places stored by the instrument and used for correcting the analyte results. Correction factors for one additional analyte shall be reported using the empty column and list the analyte's chemical symbol in the blank two-space header field provided for that column.

3.4.12.2.4   If corrections are not applied for an analyte, a zero [0] shall be entered for that analyte to indicate that the corrections were determined to be zero. Correction factors for more than one additional analyte shall be reported using Form 10B-IN.

        NOTE: Correction factors for Al, Ca, Fe, and Mg are all required and are to be listed first (as they appear on Form 10A-IN).

3.4.13   ICP-AES Interelement Correction Factors [Form 10B-IN]. This form is not required for Level 2a deliverables.

3.4.13.1   Purpose

    This form is used if correction factors for analytes other than Al, Ca, Fe, Mg, and one more analyte of the Contractor's choice were applied to the analytes analyzed by ICP-AES.

3.4.13.2   Instructions

    Complete this form following the instructions for Form 10A-IN (see Exhibit B, Section 3.4.12) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.

3.4.13.2.1   Columns of correction factors for additional analytes shall be entered left to right starting on Form 10A-IN and proceeding to Form 10B-IN, according to the alphabetical order of their chemical symbols.

3.4.14   ICP-MS Internal Standard Association [Form 11-IN]. This form is not required for Level 2a deliverables.

3.4.14.1   Purpose

    This form is used to report the associated internal standards for each target analyte for each ICP-MS instrument used in analysis.

3.4.14.2   Instructions

    Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.14.2.1   "Date", enter the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.

3.4.14.2.2   Under column "Assoc. Internal Standard 1", enter the chemical symbol of the internal standard associated with each target analyte in the analytical sequence.

3.4.14.2.3   Under column "Assoc. Internal Standard 2", if a second internal standard is used for the analyte, then enter the chemical symbol of the second internal standard associated with each target analyte in the analytical sequence. Otherwise leave blank.

3.4.15   Analysis Log [Form 12-IN]. This form is not required for Level 2a deliverables.

3.4.15.1   Purpose

    This form is used to report the analytical sequence.

3.4.15.1.1   An analytical sequence is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first SOW-required calibration standard or tune standard, and terminated by, and including, the CCV and CCB following the last SOW-required analytical sample.

3.4.15.1.2   All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, ICSs, LCSs, Preparation Blanks, LEBs, PE samples, duplicates, serial dilutions, matrix spikes, and post-digestion/distillation spikes) associated with the SDG shall be reported on Form 12-IN. The analytical sequence shall be continuous and inclusive of all analyses performed on the particular instrument during the analytical sequence.

3.4.15.1.3   Submit one Form 12-IN per analytical sequence. If more analyses were performed in the analytical sequence than will fit on one form, submit additional Form(s) 12-IN as appropriate.

3.4.15.1.4   The Analysis Logs shall be ordered chronologically. Each analytical sequence shall start on a separate Form 12-IN. Therefore, an instrument calibration or tune shall be the first entry on the form for each new analytical sequence. In addition, the analytical sequence is considered to have ended if it is interrupted for any reason, including termination for failing QC parameters.

3.4.15.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.15.2.1   "Start Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was started.

3.4.15.2.2   "End Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was ended.

3.4.15.2.3   Under column "EPA Sample No.", enter the EPA Sample Number of each analysis, including all QC operations applicable to the SDG (formatted according to Exhibit B, Table 5 – Codes for Labeling Data). All EPA Sample Numbers shall be listed in increasing chronological (date and time) order of analysis, continuing to the next Form 12-IN for the analytical sequence, if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical sequence, shall be reported. Those analyses shall be identified with the EPA Sample Number of "ZZZZZZ".

3.4.15.2.4   Under column "D/F", enter the dilution factor by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D. The dilution factor is required for all entries on Form 12-IN.

NOTE: For a particular sample, a dilution factor of "1.0" shall be entered if the digestate or distillate was analyzed without adding any further volume of dilutant.

3.4.15.2.5   For EPA-supplied solutions such as ICVs and ICSs, a dilution factor shall be entered if the supplied solution had to be diluted to a dilution different from that specified by the instructions provided with the solution. The dilution factor reported in such a case shall be that which would make the reported true values on the appropriate form for the solution equal to those that were supplied with the solution by the EPA. For instance, ICV-2(0887) has a true value of 104.0 µg/L at a 20-fold dilution. If the solution is prepared at a 40-fold dilution, a dilution factor of "2.0" shall be entered on Form 13-IN and the uncorrected instrument reading is compared to a true value of 52 µg/L. In this example, Form 2-IN will have a true value of 104.0 regardless of the dilution used. The found value for the ICV shall be corrected for the dilution listed on Form 12-IN using the following equation:

EQ. 13 ICV/CCV Correction for Dilution

$$\text{Found value on Form 2-IN} = \text{Instrument readout} \, (\mu g/L) \times D/F$$

WHERE,
D/F   =   Dilution Factor

3.4.15.2.6   Under column "Time", enter the time (in military format – HHMM) that each analysis was performed.

3.4.15.2.7   Under column "Analytes", enter the chemical symbol for each analyte reported (target and non-target) from the analytical sequence in alphabetical order by name. The Contractor is not required to report analytes (target or interferent) that are not analyzed in that analytical sequence. Enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the box empty if that analysis was not used to report the particular analyte.

3.4.15.2.7.1   Entering "X" appropriately is very important. The "X" is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Forms 1-IN – 8-IN. For each analyte result reported on any of the Forms 1-IN – 8-IN, there shall be one, and only one, properly identified entry on Form 12-IN for which an "X" is entered in the column for that analyte.

3.4.15.2.7.2   If, on Form 12-IN, an "X" is entered in the column for an analyte for a field sample associated with a dilution factor greater than 1.0, flag the data for that analyte with a "D" on the appropriate Form 1-IN.

3.4.16   ICP-MS Tune [Form 13-IN]. This form is not required for Level 2a deliverables.

3.4.16.1   Purpose

This form is used to report the tuning results for each ICP-MS instrument used in SDG analyses.

3.4.16.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.16.2.1   "Date:", enter the date (formatted as MM/DD/YYYY) the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the CSF.

3.4.16.2.2   Under column "Avg. Measured Mass (u)", enter the average mass calculated from the five or more tune integrations (in atomic mass units) measured for each isotope to one decimal point.

3.4.16.2.3   Under column "Avg. Peak Width (u)", enter the average peak width calculated from the analysis (in atomic mass units) at the percent of peak height recommended by the instrument manufacturer for each isotope to one decimal point.

3.4.16.2.4   Under column "%Height", enter the percent of peak height at which the Average Peak Width was measured to the nearest whole number.

3.4.16.2.5   Under column "%RSD", enter the percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five or more tune integrations using the following equation:

EQ. 14 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

WHERE,
SD   = Standard deviation of Tune replicates (per isotope) from EQ. 3
$\overline{X}$ = Mean value of the Tune replicates (per isotope) from EQ. 4

3.4.17   ICP-MS Internal Standards Relative Intensity Summary [Form 14-IN]. This form is not required for Level 2a deliverables.

3.4.17.1   Purpose

This form is used to report the relative internal standard intensity levels during an ICP-MS analytical sequence. The relative intensity of each of the internal standards in all analyses performed by ICP-MS must be reported on the form.

3.4.17.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.17.2.1   "Start Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was started.

3.4.17.2.2   For "End Date:", enter the date (formatted as MM/DD/YYYY) on which the analytical sequence was ended.

3.4.17.2.3   Under column "Time", enter the time (in military format – HHMM) that each analysis was performed.

3.4.17.2.4   Under column "Internal Standards %RI For:", enter the chemical symbol and elemental expression number of the internal standard in the "Element" header field provided to indicate the internal standard and elemental expression for which the Relative Intensity (RI) of the internal standards will be calculated in that column.

3.4.17.2.4.1   In the "Element" column, enter the internal standard relative intensity (to the nearest whole number) of the internal standard for each sample analysis listed on the form (excluding samples identified as "ZZZZZZ"). The internal standard percent relative intensity (%RI) is calculated using the following equation:

EQ. 15 Internal Standard Percent Relative Intensity

$$\%RI = \frac{I_n}{I_o} \times 100$$

WHERE,

I_o = The intensity of the internal standard in the blank calibration standard

I_n = The intensity of the internal standard in the EPA Sample Number

3.4.17.2.5   Under "Q" column to the right of each "Element" column, enter an "*" if the %RI for a field sample, PE, duplicate, or spike is less than 60 or greater than 125; otherwise leave the field empty.

3.4.17.2.6   Columns of internal standard RI must be entered left to right, starting with the internal standards of the lower mass on the first Form 14-IN and proceeding to the following Form 14-IN as appropriate. All Forms 14-IN for the lowest numeric instrument must be reported in ascending order by the Start Date before proceeding to the next Form 14-IN.

3.4.17.3   All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, ICSs, LCS, Preparation Blanks, LEBs, serial dilutions, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form 14-IN. The analytical sequence must be continuous and inclusive of all analyses performed on the particular instrument during the analytical sequence.

3.4.17.4   Submit one Form 14-IN per analytical sequence. If more analyses were performed in the analytical sequence, than will fit on one form, submit additional Form(s) 14-IN as appropriate. Each new analytical sequence must be started on the first line of Form 14-IN.

3.4.17.5   If more than one ICP-MS instrument or analytical sequence is used, submit additional Form(s) 14-IN as appropriate. All analytical sequences for the lowest alphanumeric instrument must be reported in ascending order before proceeding to the analytical sequences for the next highest instrument.

3.4.18   Initial Calibration [Form 15-IN]. This form is not required for Level 2a deliverables.

3.4.18.1   Purpose

This form is used to report instrument response and concentration data for each standard in the initial calibration of an instrument.

3.4.18.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.18.2.1   "Start Date:", enter the date (formatted as MM/DD/YYYY) that the calibration began.

3.4.18.2.2   Under column "True", enter the expected concentration or true amount of each analyte in the calibration standard or level. It is not required to enter a value for each analyte in every standard, so long as a value is entered for each concentration used to calibrate the instrument for the analyte.

3.4.18.2.3   Under column "Found", enter the measured concentration or amount of each analyte in the calibration standard obtained when the calibration standard was refitted to the calibration equation.

3.4.18.2.4   Under column "%D", enter the percent difference for each analyte at each concentration or amount used to calibrate the instrument other than the blank standard. Calculate the percent difference (reported to the nearest whole number) according to the following equation:

EQ. 16 Percent Difference

$$\%D = \frac{Found - True}{True} \times 100$$

WHERE,
True   = The expected concentration in the calibration standard
Found = The measured concentration in the calibration standard when the response is refitted to the calibration equation

3.4.18.2.5   Since a minimum of six levels of calibration are required (a blank plus five standards), submit a minimum of two Forms 15-IN for each calibration performed. Submit a set of Forms 15-IN for each calibration performed for each instrument used to analyze samples.

3.4.19   Initial Calibration Summary [Form 16-IN]. This form is not required for Level 2a deliverables.

3.4.19.1   Purpose

This form is used to report instrument response and concentration data for each standard in the initial calibration of an instrument.

3.4.19.2   Instructions

Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

3.4.19.2.1   For "Start Date:", enter the date (formatted as MM/DD/YYYY) that the calibration began.

3.4.19.2.2   Under column "Corr. Coeff", enter the correlation coefficient calculated for the calibration curve for each analyte calibrated in that analytical sequence to at least four significant figures.

3.4.19.2.3   Under column "Slope", enter the calculated slope of the calibration curve for each analyte calibrated in that analytical sequence to at least three significant figures.

3.4.19.2.4   Under column "Intercept", enter the calculated intercept of
              the calibration curve for each analyte calibrated in that
              analytical sequence to at least three significant figures.

3.4.19.2.5   Under column "Calib. Type", enter the calibration type for
              each analyte calibrated. Report "Lin. Reg" for linear
              regression; "LR Blank" for linear regression with blank force;
              "WLR" for weighted linear regression; or "WLR Blank" for
              weighted linear regression with blank force, as appropriate.

3.4.19.2.6   Under column "Weighting", enter the weighting factor for the
              calibration curve for each analyte calibrated in that
              analytical sequence. Report "Inverse Conc" for the inverse of
              the concentration; "Inverse Square Conc" for the inverse
              square of concentration; "Variance" for variance; "Inverse
              Variance" for the inverse of the variance; "Standard
              Deviation" for standard deviation; "Inverse Stand Dev" for the
              inverse of the standard deviation; "Inverse Square Stand Dev"
              for the inverse square of the standard deviation; or "None" if
              no weighting factor was applied.

3.4.19.2.7   Submit one set of Forms 16-IN for each calibration performed
              for each instrument used to analyze samples in the reported
              SDG.

3.5   Sample Log-In Sheet [Form DC-1]

3.5.1   Purpose

        This form is used to document the receipt and inspection of samples
        and containers. At least one original Form DC-1 is required for
        each sample shipping container (e.g., cooler). If the samples in a
        single sample shipping container must be assigned to more than one
        SDG, the original Form DC-1 shall be placed with the deliverables
        for the SDG that has the lowest alpha-numeric number and a copy of
        Form DC-1 shall be placed with the deliverables for the other
        SDG(s). The copies should be identified as "copy(ies)", and the
        location of the original should be noted on the copies.

3.5.2   Instructions

3.5.2.1   Sign and date the airbill. (If an airbill is not received,
           include a hardcopy receipt requested from the shipping company or
           a printout of the shipping company's electronic tracking
           information).

3.5.2.2   Examine the shipping container and record the presence/absence of
           custody seals and their condition (i.e., intact, broken) in
           Item 1.

3.5.2.3   Record the custody seal numbers in Item 2.

3.5.2.4   Open the container, remove the enclosed sample documentation, and
           record the presence/absence of EPA forms (i.e., TR/COC Records,
           packing lists) and airbills or airbill stickers in Items 3 and 4.
           Specify if there is an airbill present or an airbill sticker in
           Item 4. Record the airbill or sticker number in Item 5.

3.5.2.5   Remove the samples from the shipping container(s), examine the
           samples and the Sample Tags (if present), and record the
           condition of the sample bottles (i.e., intact, broken, leaking)
           and presence or absence of Sample Tags in Items 6 and 7.

3.5.2.6   Record the presence or absence of a shipping container
           temperature indicator bottle in Item 8.

3.5.2.7   Record the shipping container temperature in Item 9. If ice is
           present, that shall be noted in the "Remarks" column.

3.5.2.8   Review the sample shipping documents and compare the information
           recorded on all the documents and samples and mark the
           appropriate answer in Item 10.

3.5.2.9   The log-in date should be recorded at the top of Form DC-1;
           record the date and time of shipping container receipt at the
           laboratory in Items 11 and 12.

3.5.2.10   If there are no problems observed during receipt, sign and date
           (include the time) Form DC-1 and the TR/COC Record, and write the
           sample numbers in the "EPA Sample #" column.

3.5.2.11   Record the pH for all aqueous/water samples received.

3.5.2.12   Record the appropriate Sample Tags and assigned laboratory
           numbers, if applicable.

3.5.2.13   Any comments should be made in the "Remarks" column.

3.5.2.14   For Items 1, 3, 4, 6, 7, 8, and 10, circle the appropriate
           response. Responses can be underlined if this form is completed
           by automated equipment. Unused columns and spaces shall be
           crossed out, initialed, and dated.

3.5.2.15   If there are problems observed during receipt (including samples
           that have not been preserved to the proper pH) or an answer
           marked with an asterisk (e.g., "absent*") was circled, contact
           SMO and document the contact as well as resolution of the problem
           on a CLP Communication Log and in the SDG Narrative. Following
           resolution, sign and date the forms as specified in the preceding
           paragraph and note, where appropriate, the resolution of the
           problem.

3.6   Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]

3.6.1   Purpose

         The CSF Inventory Sheet is used to record both the inventory of CSF
         documents and the number of documents in the original Sample Data
         Package which is sent to the EPA Region.

3.6.2   Instructions

3.6.2.1   Organize all EPA-CSF documents as described in Exhibit B,
           Sections 2.0 and 3.0. Assemble the documents in Exhibit B,
           Section 2.0 in the order specified on Form DC-2, and stamp each
           page with the consecutive number. Inventory the CSF by reviewing
           the document numbers and recording page number ranges in the
           columns provided on Form DC-2. The Contractor shall verify and
           record in the "Comments" section on Form DC-2 all intentional
           gaps in the page numbering sequence (for example, "page numbers
           not used, XXXX-XXXX, XXXX-XXXX"). If there are no documents for
           a specific document type, enter an "NA" in the empty space.

3.6.2.2   Certain laboratory-specific documents related to the CSF may not
           fit into a clearly defined category. The laboratory should
           review Form DC-2 to determine if it is most appropriate to place
           them under Categories 66 through 68. Category 68 should be used
           if there is no appropriate previous category. These types of
           documents should be described or listed in the blanks under each
           appropriate category.

3.6.2.3   If it is necessary to insert new or inadvertently omitted
           documents, the Contractor shall follow these steps:

- Number all documents to be inserted with the next sequential
  numbers and file the inserts in their logical positions within
  the CSF (e.g., document to be inserted between pages 6 and 7
  shall be numbered as 6a, 6b, 6c, etc.). Identify where the
  inserts are filed in the CSF by recording the document numbers
  and their locations under the "Other Records" section of Form
  DC-2 (e.g., documents to be inserted between pages 6 and 7
  shall be numbered as 6a, 6b, 6c, etc.).

4.0   DATA REPORTING FORMS

       The data reporting forms are shown on the following pages.

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EXHIBIT B

INORGANIC FORMS

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SDG COVER PAGE

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

SOW No.: _____

			Analysis Method		
EPA Sample No.	Lab Sample ID	ICP-AES	ICP-MS	Mercury	Cyanide
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed in the SDG Narrative. Release of the data contained in this hardcopy Complete SDG File and in the electronic data submitted has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: _____   Name: _____

Date: _____   Title: _____

EPA SAMPLE NO.

FORM 1-IN
INORGANIC ANALYSIS DATA SHEET

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Matrix: _____   Lab Sample ID: _____

% Solids: _____   Date Received: _____

Analytical Method: _____

Concentration Units (μg/L, mg/L, mg/kg dry weight or μg): _____

CAS No.	Analyte	Concentration	Q	Date Analyzed	Time Analyzed

NOTE: Hardness (total) is reported in mg/L

Comments:
_____

_____

_____

FORM 2-IN

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Initial Calibration Verification Source: _____

Continuing Calibration Verification Source: _____

Run Batch: _____ Analytical Method: _____

Concentration Units: µg/L

| | Initial Calibration Verification | | | | Continuing Calibration Verification | | | | | | |
| | ID: | | | | ID: | | | | ID: | | |
Analyte	True	Found	%R	%RSD	True	Found	%R	%RSD	Found	%R	%RSD

```
                              FORM 3-IN
                               BLANKS
```

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Preparation Blank Matrix: _____

Preparation Blank Concentration Units (µg/L, mg/L, mg/kg dry weight, or µg): _____

Analytical Method: _____ Preparation Batch: _____

Run Batch: _____ Preparation Method: _____

Analyte	Initial Calibration Blank (ug/L)		Continuing Calibration Blank (ug/L)								Preparation Blank/Leachate Extraction Blank	
	ID:	Q	ID:	Q	ID:	Q	ID:	Q	ID:	Q	ID:	Q

```
                           FORM 4-IN
                   ICP INTERFERENCE CHECK SAMPLE
```

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Analytical Method: _____   ICSA Source: _____

Instrument ID: _____   ICSB Source: _____

Run Batch: _____

Concentration Units: µg/L

Analyte	True		Found			
	ICSA	ICSAB	ICSA	%R	ICSAB	%R

EPA SAMPLE NO.

FORM 5A-IN

MATRIX SPIKE SAMPLE RECOVERY

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Matrix: _____   Analytical Method: _____

% Solids: _____

Concentration Units (µg/L, mg/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q

EPA SAMPLE NO.

FORM 5B-IN

POST-DIGESTION/DISTILLATION SPIKE

SAMPLE RECOVERY

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Matrix: _____   Analytical Method: _____

% Solids: _____

Concentration Units (µg/L, mg/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	Q	Sample Result (SR)	Q	Spike Added (SA)	%R	Q

EPA SAMPLE NO.

FORM 6-IN

DUPLICATES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Matrix: _____ Analytical Method: _____

% Solids: _____

Concentration Units (µg/L, mg/L, or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	Q	Duplicate (D)	Q	RPD	Q

EPA SAMPLE NO.

FORM 7-IN

LABORATORY CONTROL SAMPLE

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Matrix: _____   Preparation Method: _____

Analytical Method: _____   Preparation Batch: _____

Concentration Units (µg/L, mg/L, mg/kg dry weight, or µg): _____

Analyte	True	Found	%R

EPA SAMPLE NO.

FORM 8-IN

ICP-AES AND ICP-MS SERIAL DILUTIONS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Matrix: _____ Analytical Method: _____

% Solids: _____

Concentration Units (µg/L, mg/L or mg/kg dry weight): _____

Analyte	Initial Sample Result (I)	Q	Serial Dilution Result (S)	Q	% Difference	Q

FORM 9-IN
METHOD DETECTION LIMIT

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Analytical Method: _____ Instrument ID: _____

Preparation Method: _____

Concentration Units (µg/L, µg, or mg/kg): _____

Analyte	Wavelength/Mass	MDL	Date Analyzed

FORM 10A-IN
ICP-AES INTERELEMENT CORRECTION FACTORS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		Al	Ca	Fe	Mg	____

FORM 10B-IN
ICP-AES INTERELEMENT CORRECTION FACTORS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Instrument ID: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		_____	_____	_____	_____	_____

```
FORM 11-IN
ICP-MS INTERNAL STANDARD ASSOCIATION
```

Lab Name: _____ Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Instrument ID: _____   Date: _____

Run Batch: _____

Analyte	Assoc. Internal Standard 1	Assoc. Internal Standard 2

```
FORM 11-IN
```

```
FORM 12-IN
ANALYSIS LOG
```

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ MA No.: _____ SDG No.: _____

Instrument ID: _____ Analytical Method: _____

Start Date: _____ End Date: _____

Run Batch: _____

EPA Sample No.	D/F	Time	Analytes																								

FORM 13-IN
ICP-MS TUNE

EPA SAMPLE NO.

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Instrument ID: _____   Date: _____

Run Batch: _____

Element - Mass	Avg. Measured Mass (u)	Average Peak Width (u)	%Height	%RSD
Be – 9				
Mg – 24				
Mg – 25				
Mg – 26				
Co – 59				
In – 113				
In – 115				
Pb – 206				
Pb – 207				
Pb – 208				

FORM 14-IN
ICP-MS INTERNAL STANDARDS RELATIVE INTENSITY SUMMARY

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Instrument ID: _____   Start Date: _____

Run Batch: _____   End Date: _____

EPA Sample No.	Time	Internal Standards %RI For:									
		Element	Q	Element	Q	Element	Q	Element	Q	Element	Q

```
FORM 15-IN
INITIAL CALIBRATION
```

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Instrument ID: _____   Start Date: _____

Analytical Method: _____   Run Batch: _____

Concentration Units: µg/L

Analyte	True	Found	%D	True	Found	%D	True	Found	%D

FORM 16-IN
INITIAL CALIBRATION SUMMARY

Lab Name: _____   Contract: _____

Lab Code: _____   Case No.: _____   MA No.: _____   SDG No.: _____

Instrument ID: _____   Start Date: _____

Analytical Method: _____   Run Batch: _____

Analyte	Corr. Coeff.	Slope	Intercept	Calib. Type	Weighting

FORM 16-IN
INITIAL CALIBRATION SUMMARY

FORM DC-1
SAMPLE LOG-IN SHEET

Lab Name		Page of
Received By (Print Name)		Log-in Date
Received By (Signature)		
Case Number	SDG No.	MA No.

Remarks:			EPA Sample #	Aqueous/ Water Sample pH	Corresponding		Remarks: Condition of Sample Shipment, etc.
					Sample Tag #	Assigned Lab #	
1. Custody Seal(s)	Present/Absent* Intact/Broken	1					
2. Custody Seal Nos.	_____ _____	2					
3. Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*	3					
		4					
4. Airbill	Airbill/Sticker Present/Absent*	5					
5. Airbill No.	_____ _____	6					
6. Sample Tags Sample Tag Numbers	Present/Absent* Listed/Not Listed on Traffic Report/Chain of Custody Record	7					
		8					
		9					
7. Sample Condition	Intact/Broken*/ Leaking	10					
8. Shipping Container Temperature Indicator Bottle	Present/Absent*	11					
		12					
		13					
9. Shipping Container Temperature	_____	14					
10. Does information on Traffic Reports/Chain of Custody Records and Sample Tags agree?	Yes/No*	15					
		16					
		17					
		18					
11. Date Received at Lab	_____	19					
12. Time Received	_____	20					
		21					
		22					

* Contact SMO and attach record of resolution

Reviewed By	Logbook No.
Date	Logbook Page No.

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

LAB NAME _____

LAB CODE _____

CONTRACT NO. _____

CASE NO. _____ SDG NO. _____

MA NO. _____

SOW NO. _____

All documents delivered in the Complete SDG File must be original documents where possible. (Reference - Exhibit B Section 2.4)

| | | PAGE NOs. | | CHECK | |
		FROM	TO	LAB	REGION
1.	SDG Cover Page				
2.	Traffic Report/Chain of Custody Record(s)				
3.	Sample Log-In Sheet (DC-1)				
4.	CSF Inventory Sheet (DC-2)				
5.	SDG Narrative				

Inorganic Analysis

ICP-AES

6.	Inorganic Analysis Data Sheet (Form 1-IN)				
7.	Initial and Continuing Calibration Verification (Form 2-IN)				
8.	Blanks (Form 3-IN)				
9.	ICP Interference Check Sample (Form 4-IN)				
10.	Matrix Spike Sample Recovery (Form 5A-IN)				
11.	Post-Digestion/Distillation Spike Sample Recovery (Form 5B-IN)				
12.	Duplicates (Form 6-IN)				
13.	Laboratory Control Sample (Form 7-IN)				
14.	ICP-AES and ICP-MS Serial Dilutions (Form 8-IN)				
15.	Method Detection Limit (Form 9-IN)				
16.	ICP-AES Interelement Correction Factors (Form 10A-IN)				
17.	ICP-AES Interelement Correction Factors (Form 10B-IN)				
18.	Analysis Log (Form 12-IN)				
19.	Initial Calibration (Form 15-IN)				

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

		PAGE NOs.		CHECK	
		FROM	TO	LAB	REGION
20.	Initial Calibration Summary (Form 16-IN)	_____	_____	_____	_____
21.	ICP-AES Raw Data	_____	_____	_____	_____
22.	ICP-AES Preparation Log Books, Preparation records, Analysis records, and PE Instructions	_____	_____	_____	_____
ICP MS					
23.	Inorganic Analysis Data Sheet (Form 1-IN)	_____	_____	_____	_____
24.	Initial and Continuing Calibration Verification (Form 2-IN)	_____	_____	_____	_____
25.	Blanks (Form 3-IN)	_____	_____	_____	_____
26.	ICP Interference Check Sample (Form 4-IN)	_____	_____	_____	_____
27.	Matrix Spike Sample Recovery (Form 5A-IN)	_____	_____	_____	_____
28.	Post-Digestion/Distillation Spike Sample Recovery (Form 5B-IN)	_____	_____	_____	_____
29.	Duplicates (Form 6-IN)	_____	_____	_____	_____
30.	Laboratory Control Sample (Form 7-IN)	_____	_____	_____	_____
31.	ICP-AES and ICP-MS Serial Dilutions (Form 8-IN)	_____	_____	_____	_____
32.	Method Detection Limit (Form 9-IN)	_____	_____	_____	_____
33.	ICP-MS Internal Standard Association (Form 11-IN)	_____	_____	_____	_____
34.	Analysis Log (Form 12-IN)	_____	_____	_____	_____
35.	ICP-MS Tune (Form 13-IN)	_____	_____	_____	_____
36.	ICP-MS Internal Standards Relative Intensity Summary (Form 14-IN)	_____	_____	_____	_____
37.	Initial Calibration (Form 15-IN)	_____	_____	_____	_____
38.	Initial Calibration Summary (Form 16-IN)	_____	_____	_____	_____
39.	ICP-MS Raw Data	_____	_____	_____	_____
40.	ICP-MS Preparation Log Books, Preparation records, Analysis records, and PE Instructions	_____	_____	_____	_____
Mercury					
41.	Inorganic Analysis Data Sheet (Form 1-IN)	_____	_____	_____	_____
42.	Initial and Continuing Calibration Verification (Form 2-IN)	_____	_____	_____	_____
43.	Blanks (Form 3-IN)	_____	_____	_____	_____
44.	Matrix Spike Sample Recovery (Form 5A-IN)	_____	_____	_____	_____
45.	Duplicates (Form 6-IN)	_____	_____	_____	_____
46.	Method Detection Limit (Form 9-IN)	_____	_____	_____	_____
47.	Analysis Log (Form 12-IN)	_____	_____	_____	_____
48.	Initial Calibration (Form 15-IN)	_____	_____	_____	_____

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

49. Initial Calibration Summary (Form 16-IN)   _____ _____ _____ _____

50. Mercury Raw Data   _____ _____ _____ _____

51. Mercury Preparation Log Books, Preparation
     records, Analysis records, and PE Instructions   _____ _____ _____ _____

Cyanide

52. Inorganic Analysis Data Sheet (Form 1-IN)   _____ _____ _____ _____

53. Initial and Continuing Calibration Verification
     (Form 2-IN)   _____ _____ _____ _____

54. Blanks (Form 3-IN)   _____ _____ _____ _____

55. Matrix Spike Sample Recovery (Form 5A-IN)   _____ _____ _____ _____

56. Post-Digestion/Distillation Spike Sample
     Recovery (Form 5B-IN)   _____ _____ _____ _____

57. Duplicates (Form 6-IN)   _____ _____ _____ _____

58. Method Detection Limit (Form 9-IN)   _____ _____ _____ _____

59. Analysis Log (Form 12-IN)   _____ _____ _____ _____

60. Initial Calibration (Form 15-IN)   _____ _____ _____ _____

61. Initial Calibration Summary (Form 16-IN)   _____ _____ _____ _____

62. Cyanide Raw Data   _____ _____ _____ _____

63. Cyanide Preparation Log Books, Preparation
     records, Analysis records, and PE Instructions   _____ _____ _____ _____

Additional

64. Percent Solids Determination Log   _____ _____ _____ _____

65. EPA Shipping/Receiving Documents   _____ _____ _____ _____

     Airbill (No. of Shipments _____)   _____ _____ _____ _____

     Sample Tags   _____ _____ _____ _____

     Sample Log-In Sheet (Lab)   _____ _____ _____ _____

66. Misc. Shipping/Receiving Records
     (list all individual records)
     Communication Logs

     _____   _____ _____ _____ _____

     _____

67. Internal Lab Sample Transfer Records &
     Tracking Sheets (describe or list)   _____ _____ _____ _____

     _____

     _____

68. Other Records (describe or list)   _____ _____ _____ _____
     Communication Logs

     _____   _____ _____ _____ _____

     _____

FORM DC-2
FULL INORGANICS COMPLETE SDG FILE (CSF) INVENTORY SHEET

69. Comments:
_____

_____

_____

Completed by:
(CLP Lab) _____   _____   _____
          (Signature)   (Print Name & Title)   (Date)

Audited by:
(EPA)   _____   _____   _____
          (Signature)   (Print Name & Title)   (Date)

EXHIBIT C

INORGANIC TARGET ANALYTE LIST AND
CONTRACT REQUIRED QUANTITATION LIMITS

NOTE: The Contract Required Quantitation Limit (CRQL) values listed on the
following pages are based on the analysis of samples according to the
specifications given in Exhibit D.

Changes to the CRQL may be requested under the Modified Analysis (MA)
clause in the contract.

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Exhibit C - Inorganic Target Analyte List and Contract
Required Quantitation Limits

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1.0   ICP-AES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 1. ICP-AES TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A]

Analyte Name	CAS Number	CRQL Water[D] (μg/L)	Soil[B] (mg/kg)	Wipe (μg)	TCLP (mg/L)
Aluminum	7429-90-5	200	20	20	--
Antimony	7440-36-0	60	6	6	--
Arsenic	7440-38-2	10	1	1	5
Barium	7440-39-3	200	20	20	100
Beryllium	7440-41-7	5	0.5	0.5	--
Cadmium	7440-43-9	5	0.5	0.5	1
Calcium	7440-70-2	5000	500	500	--
Chromium	7440-47-3	10	1	1	5
Cobalt	7440-48-4	50	5	5	--
Copper	7440-50-8	25	2.5	2.5	--
Iron	7439-89-6	100	10	10	--
Lead	7439-92-1	10	1	1	5
Magnesium	7439-95-4	5000	500	500	--
Manganese	7439-96-5	15	1.5	1.5	--
Nickel	7440-02-0	40	4	4	--
Potassium	7440-09-7	5000	500	500	--
Selenium	7782-49-2	35	3.5	3.5	1
Silver	7440-22-4	10	1	1	5
Sodium	7440-23-5	5000	500	500	--
Thallium	7440-28-0	25	2.5	2.5	--
Vanadium	7440-62-2	50	5	5	--
Zinc	7440-66-6	60	6	6	--
Hardness (total)	Hardness	33[C]	--	--	--

2.0   ICP-MS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 2. ICP-MS TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[A]

Analyte Name	CAS Number	CRQL Water (µg/L)	Soil[B] (mg/kg)
Aluminum	7429-90-5	20	--
Antimony	7440-36-0	2	1
Arsenic	7440-38-2	1	0.5
Barium	7440-39-3	10	5
Beryllium	7440-41-7	1	0.5
Cadmium	7440-43-9	1	0.5
Calcium	7440-70-2	500	--
Chromium	7440-47-3	2	1
Cobalt	7440-48-4	1	0.5
Copper	7440-50-8	2	1
Iron	7439-89-6	200	--
Lead	7439-92-1	1	0.5
Magnesium	7439-95-4	500	--
Manganese	7439-96-5	1	0.5
Nickel	7440-02-0	1	0.5
Potassium	7440-09-7	500	--
Selenium	7782-49-2	5	2.5
Silver	7440-22-4	1	0.5
Sodium	7440-23-5	500	--
Thallium	7440-28-0	1	0.5
Vanadium	7440-62-2	5	2.5
Zinc	7440-66-6	2	1

3.0   MERCURY BY COLD VAPOR ATOMIC ABSORPTION TARGET ANALYTE LIST AND
      CONTRACT REQUIRED QUANTITATION LIMITS

TABLE 3. MERCURY BY COLD VAPOR ATOMIC ABSORPTION TARGET ANALYTE LIST AND
CONTRACT REQUIRED QUANTITATION LIMITS

Analyte Name	CAS Number	CRQL Water[D] (µg/L)	Soil[B] (mg/kg)	TCLP (mg/L)
Mercury	7439-97-6	0.2	0.1	0.2

4.0   CYANIDE BY SPECTROPHOTOMETRY TARGET ANALYTE LIST AND CONTRACT REQUIRED
      QUANTITATION LIMITS

TABLE 4. CYANIDE BY SPECTROPHOTOMETRY TARGET ANALYTE LIST AND CONTRACT
REQUIRED QUANTITATION LIMITS

Analyte Name	CAS Number	CRQL Water[D] (µg/L)	Soil[B] (mg/kg)
Cyanide	57-12-5	10	0.5

Endnotes:

A. Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) may be requested under the Modified Analysis clause in the contract.

B. The CRQLs for soil/sediment are based on 100% solids and on the minimum weights and volumes specified in Exhibit D. The moisture content of the samples must be used to adjust the CRQL values appropriately.

C. Hardness (total) is reported as a calculation in mg/L.

D. Use the water CRQLs for Synthetic Precipitation Leaching Procedure (SPLP).

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EXHIBIT D

INTRODUCTION TO INORGANIC ANALYTICAL METHODS

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Exhibit D – Introduction to Inorganic Analytical Methods

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1.0   INTRODUCTION

The inorganic analytical service provides a contractual framework for laboratories. This framework applies the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in aqueous/water, soil/sediment, and 22 metals (excluding mercury) in wipe samples.

The analytical methods that follow are designed to analyze aqueous/water, leachate, soil/sediment, and wipe samples from hazardous waste sites for the presence of inorganic analytes contained in the Inorganic Target Analyte List and Contract Required Quantitation Limits (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits). The inorganic methods include multiple preparation procedures and Quality Control (QC) requirements. Analytical techniques in the inorganic methodologies include Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), Cold Vapor Atomic Absorption Spectroscopy (CVAA), and Spectrophotometry.

2.0   INORGANIC METHODS FLOW CHART

Figure 1 outlines the general analytical scheme the Contractor shall follow in performing standard trace metals and cyanide analyses under this contract.

Figure 1 – Inorganic Methods Flow Chart
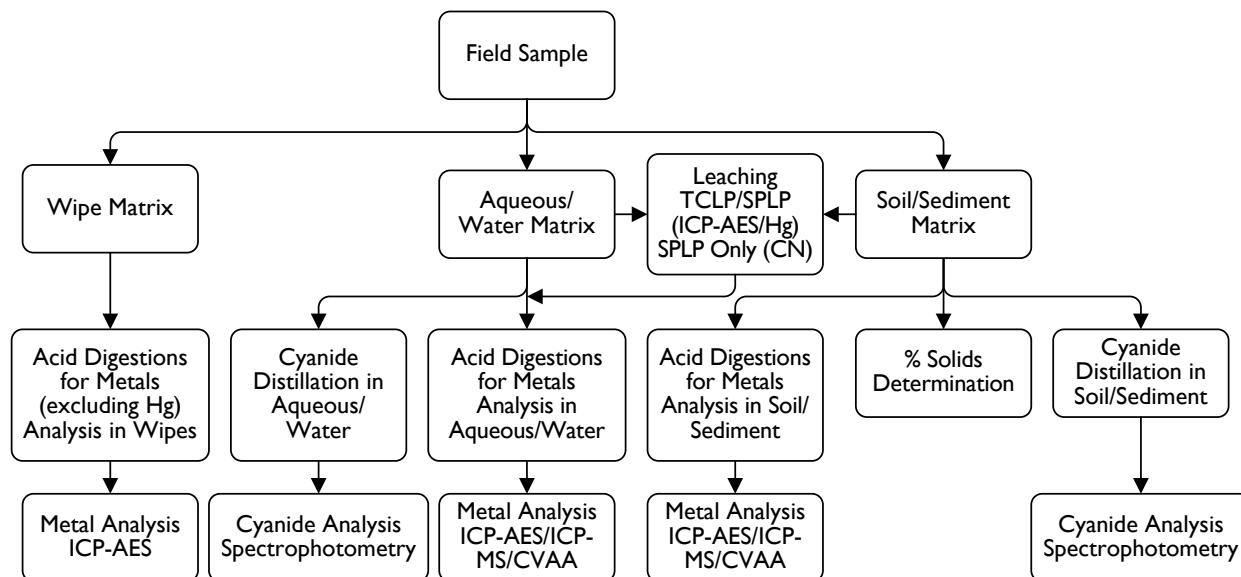
3.0   GLASSWARE CLEANING

Laboratory glassware to be used within the metals and cyanide analyses must be acid cleaned according to the EPA's manual, Methods for Chemical Analysis of Water and Wastes (EPA/600/4-79/020) or an equivalent procedure. Equivalent procedures are those which meet the Preparation Blank requirements in the Statement of Work (SOW). An electronic version of this manual can be found via the EPA's National Service Center for Environmental Publications (NSCEP) website at https://www.epa.gov/nscep (search on EPA Manual 600479020).

4.0   STANDARD STOCK SOLUTIONS

Stock solutions to be used for preparing instrument or method standards may be purchased or prepared as described in the individual methods of Exhibit D. Stock solutions that are past the manufacturer's expiration date shall not be used to prepare analytical standards.

5.0   VERIFICATION OF AQUEOUS/WATER SAMPLE PRESERVATION

5.1   At the time of sample receipt, the Contractor shall check the pH of the sample and note in a sample receipt log if the pH is less than or equal to 2 for metals or is greater than or equal to 10 for a cyanide sample.

5.1.1   If a metals sample has not been properly preserved, the Contractor shall adjust the pH of the sample(s) for metals (according to procedures in Exhibit D) and note this in the Sample Delivery Group (SDG) Narrative.

5.1.2   The Contractor shall not adjust the pH of a sample for cyanide. If the pH of a cyanide sample is <10, contact the Sample Management Office (SMO) for further instructions before proceeding with the preparation and analysis.

5.1.3   The Contractor shall not adjust the pH of samples scheduled for Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction.

6.0   SAMPLE CHARACTERIZATION

6.1   If multiphase samples (e.g., two-phase liquid sample, oily sludge/sandy soil/sediment sample) are received by the Contractor, the Contractor shall contact SMO to apprise them of the type of sample received. SMO will contact the EPA Region.

6.1.1   If all phases of the sample are amenable to analysis, the EPA Region may require the Contractor to do any of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.

- Separate the phases of the sample and analyze one or more of the phases, separately. SMO will provide the EPA Sample Numbers for the additional phases, if required.

- Do not analyze the sample.

6.1.2   If all of the phases are not amenable to analysis (i.e., outside scope), the EPA Region may require the Contractor to do any of the following:

- Separate the phases and analyze the phase(s) that is (are) amenable to analysis. SMO will provide the EPA Sample Numbers for the additional phases, if required.

- Do not analyze the sample.

6.1.3   The Contractor shall document the EPA Region's decision in the SDG Narrative.

7.0   SAMPLE DILUTIONS

7.1   All samples for multi-analyte analysis shall be analyzed undiluted, unless the dilution-adjusted detection limits for all analytes are below the Contract Required Quantitation Limits (CRQLs).

7.2   Samples analyzed by ICP-MS (according to procedures in Exhibit D – Inductively Coupled Plasma - Mass Spectrometry) may be analyzed at initial dilution if the results of a screening analysis indicate that this is necessary.

7.3   When an analyte concentration exceeds the calibrated range, appropriate dilution (but not below the CRQL) and reanalysis is required. The Contractor shall use the least dilution necessary to bring the analyte(s) instrument reading within the upper 75% of the calibrated range and report the highest valid value for each analyte as measured from the undiluted and diluted analyses. Unless the Contractor can submit proof that dilution was required to obtain valid results, or to avoid damage to ICP-MS instruments, both diluted and undiluted sample measurements must be contained in the raw data.

7.4   For single analyte analysis, a diluted sample analysis may be the only sample analysis performed if the analyte's instrument result is either greater than fifty (50) times the CRQL, or is in the upper 75% of the calibrated range. An undiluted sample analysis does not have to be performed in this case. The sample and its associated matrix spike and/or duplicate shall initially be analyzed at the same dilution.

7.5   All sample dilutions shall be made with reagent water mixed with the appropriate acid(s) (metals) or base (cyanide) to be consistent with the acid or base concentration in the digestate or distillate.

8.0   DISSOLVED METALS

If dissolved metals are requested by the EPA Regional Offices, the Contractor shall digest the field-filtered samples according to the procedures in Exhibit D of the analytical method and report as dissolved metals.

9.0   REPLICATE INTEGRATIONS/EXPOSURES

If the Contractor analyzes samples using multiple integrations/exposures, the Contractor must use the data obtained from all integrations/exposures to calculate the final sample result even if more than the minimum number of integrations/exposures are taken.

10.0   RAW DATA REQUIREMENTS

The Contractor is reminded and cautioned that the collection and reporting of raw data may or may not be referred to within the individual methods of Exhibit D or the Quality Assurance (QA) protocol of Exhibit F – Programmatic Quality Assurance/Quality Control Elements. The raw data deliverable requirements are specified in Exhibit B – Reporting and Deliverables Requirements, Section 2.4. Raw data collected and provided in association with the performance of analyses under this contract shall conform to the appropriate sections of Exhibit B.

11.0   ANALYTICAL STANDARDS REQUIREMENTS

The EPA will not supply analytical reference standards for either direct analytical measurements or the purpose of traceability. All contract laboratories shall be required to prepare, from materials or purchase from private chemical supply companies, those standards necessary to successfully and accurately perform the analyses required in this protocol.

11.1 Preparation of Chemical Standards from the Neat High Purity Bulk Material

11.1.1   If the laboratory cannot obtain analytical reference standards, the laboratory may prepare its own chemical standards. Laboratories shall obtain the highest purity possible when purchasing chemical standards. Standards purchased at less than 97% purity shall be documented as to why a higher purity could not be obtained.

11.1.2   The chemical standards shall be kept at manufacturer recommended conditions when not being used in the preparation of standard solutions. Proper storage of chemicals is essential to safeguard them from decomposition.

11.1.3   The Contractor is responsible for having analytical documentation demonstrating that the purity of each chemical is correctly stated. Purity confirmation, when performed, should use appropriate techniques. Use of two or more independent methods is recommended. The correction factor for impurity when weighing neat materials in the preparation of solution standards is determined using the following equation:

EQ. 1 Weight of Impure Compound

$$\text{Weight of Impure Chemical} = \frac{\text{weight of pure chemical}}{(\text{percent purity}/100)}$$

WHERE,
Weight of Pure Chemical   =   That required to prepare a specific volume of a solution standard of a specified concentration.

11.1.4   Logbooks are to be kept for all weighing and dilutions of standards and reagents. All subsequent dilutions from the primary standard and the calculations for determining their concentrations are to be reviewed and verified by a second person.

11.1.5   All solution standards are to be refrigerated, if required, when not in use.

11.1.6   All solution standards are to be clearly labeled to include the identity of the analyte or analytes, concentration, the standard ID number of the solution, date prepared, solvent, expiration date of the solution, special storage requirements (if any), and initials of the preparer.

11.2 Purchase of Chemical Standards Already in Solution

Solutions of analytical reference standards can be purchased by Contractors provided they meet the following criteria.

11.2.1   Contractors shall maintain documentation of the purity confirmation of the material to verify the integrity of the standard solutions they purchase.

11.2.2   The quality of the reference standards purchased shall be demonstrated statistically and analytically by a method of the supplier's choice.

11.3   Documentation of the Verification and Preparation of Chemical Standards

It is the responsibility of the Contractor to maintain the necessary documentation to show that the chemical standards used in the performance of the CLP analysis conform to the requirements previously listed.

11.3.1   In those cases where the documentation is supportive of the analytical results of data packages sent to the Government, such documentation is to be kept on-file by the Contractor for a period of one year.

11.3.2   Upon request by the EPA Regional CLP Contracting Officer's Representative (COR), the Contractor shall submit their most recent previous year's documentation (12 months) for the verification and preparation of chemical standards within 14 days of receipt of the request to the designated recipients.

12.0   SAFETY

The toxicity or carcinogenicity of each reagent used in this SOW has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The Contractor is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of chemicals specified in these methods. A reference file of Material Safety Data Sheets (MSDS) shall be made available to all personnel involved in the chemical analysis.

13.0   POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

14.0   WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with applicable environmental rules and regulations.

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EXHIBIT D

GENERAL INORGANIC ANALYSIS

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Exhibit D - General Inorganic Analysis

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1.0   SCOPE AND APPLICATION

This Exhibit provides procedures for the use of General Analysis to determine the percent solids of soil/sediment samples, pH, and the leaching of samples by Toxicity Characteristic Leaching Procedure (TCLP) (SW-846 Method 1311) or Synthetic Precipitation Leaching Procedure (SPLP) (SW-846 Method 1312).

2.0   SUMMARY OF METHOD

These methods describe the determination of sample characteristics by gravimetry, electrometry, or the leaching of samples for subsequent analysis by the other analytical methods in this Statement of Work (SOW).

3.0   DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0   INTERFERENCES

4.1   pH Determination

4.1.1   Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions with a pH <1 may give incorrectly high pH measurements.

4.1.2   Coatings of oily material or particulate matter can impair electrode response. These coatings can generally be removed by gentle wiping or detergent washing followed by rinsing with reagent water. Treatment with 10% HCl may be necessary to remove some films.

4.1.3   Temperature changes can affect measurements. This can be minimized by use of instruments with temperature compensation. The temperature of the sample can change the sample pH. The temperature at which pH measurements are carried out shall be noted.

5.0   SAFETY

See Section 12.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

6.0   EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this SOW is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1   Percent Solids Determination

6.1.1   Disposable weigh boats with covers

6.1.2   Oven capable of maintaining a temperature of 105°C (±5°C). Oven shall be in a well-ventilated area.

6.1.3   Balance – Top loader, 300 grams (g) capacity with a minimum sensitivity of ±1.0 milligrams (mg)

The balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each balance and be accurate to ±1.0 mg. The masses that are used to check the balances daily must be checked on a monthly basis using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '1' or '2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified every five years or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates that these criteria have been met.

6.2   pH Determinations

6.2.1   pH meter with reference electrode accurate to at least ±0.05 pH units. The pH meter/probe should be equipped with a means of temperature compensation either manually or automatically.

6.2.2   pH paper, wide-range or narrow-range pH paper strip.

6.2.3   Magnetic stirrer with fluoropolymer-coated stir bar.

6.2.4   Beakers – Preferably polyethylene or polytetrafluoroethylene (PTFE).

6.2.5   Various volumetric flasks (Class A) and calibrated pipettes. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.2.6   Thermometer that covers a range of the sample temperature with a minimum accuracy of ±1°C.

6.3   TCLP and SPLP Leaching

6.3.1   Agitation Apparatus – Capable of rotating the extraction vessel(s) in an end-over-end fashion at 30 ±2 rpm.

6.3.2   Extraction Vessels – Jar with sufficient capacity to hold sample and extraction fluid. Vessels may be constructed of PTFE, high-density polyethylene, polypropylene, polyvinyl chloride, or other suitable inert material. It is recommended that borosilicate glass bottles be used instead of other types of glass for the analysis of inorganic constituents.

6.3.3   Filters – Pre-filters must not be used. Borosilicate glass with no binder material with an effective pore size of 0.6–0.8 micrometers (µm). Acid wash with 1N nitric acid prior to use, followed by three consecutive rinses with reagent water [a minimum of 1 Liter (L) per rinse is recommended]. Glass fiber filters are fragile and should be handled with care.

6.3.4   Filtration Device – Capable of exerting pressures up to 50 psi. Recommend use of units having an internal volume of 1.5 L and capable of accommodating a 142 millimeter (mm) filter.

6.3.5   Beaker – 500 milliliters (mL).

6.3.6   Balance – Any laboratory balance accurate to within ±0.01 grams may be used (all weight measurements are to be within ±0.1 grams). All requirements in Section 6.1.3 shall be met.

6.3.7   pH meter with reference electrode accurate to at least ±0.05 units at 25°C. The pH meter/probe should be equipped with a means of temperature compensation either manually or automatically.

6.3.8   Magnetic stirrer with fluoropolymer-coated stir bar.

7.0   REAGENTS AND STANDARDS

7.1   Reagents

7.1.1   Reagent water - The purity of this water must be equivalent to ASTM
Type II water (ASTM D1193-06). Use this water for all reagents,
standards, and dilutions. For the preparation of pH buffer
solutions, it may be necessary to boil and cool the water prior to
use.

7.1.2   Hydrochloric acid, (1N) - Add 83.5 mL conc. hydrochloric acid,
32-38% (specific gravity 1.19) to 400 mL reagent water and dilute to
1 L.

7.1.3   Nitric acid, (1N) – Add 62 mL conc. nitric acid, 67-70% (specific
gravity 1.41) to 400 mL reagent water and dilute to 1 L.

7.1.4   Sodium Hydroxide, (1N) - Add 40 g reagent grade NaOH to 400 mL
reagent water and dilute to 1 L.

7.1.5   Glacial Acetic Acid – reagent grade.

7.1.6   Sulfuric Acid/Nitric Acid, (60/40 weight percent mixture) –
Cautiously mix 60 g (approximately 33 mL) of conc. sulfuric acid,
95-98% (specific gravity 1.84) with 40 g (approximately 28 mL) conc.
nitric acid. The Contractor may prepare a more diluted version of
this reagent for ease in adjusting extraction fluid pH.

7.1.7   Extraction Fluids

Extraction fluids should be monitored for impurities and the pH
checked prior to use. If impurities are found or the pH is not
within specifications, the fluid shall be discarded and fresh
extraction fluid prepared. Solutions are unbuffered and exact pH
may not be attained.

7.1.7.1   TCLP Extraction Fluid #1 – Add 5.7 mL of glacial acetic acid to
500 mL of reagent water, add 64.3 mL of 1N NaOH solution, and
dilute to 1 L. The pH of this solution should be 4.93 ±0.05.

7.1.7.2   TCLP Extraction Fluid #2 – Dilute 5.7 mL of glacial acetic acid
with reagent water to a final volume of 1 L. The pH of this
solution should be 2.88 ±0.05.

7.1.7.3   SPLP Extraction Fluid #1 – Use this solution with samples from
east of the Mississippi River. Add sufficient 60/40
Sulfuric/Nitric acid solution to reagent water until the pH is
4.20 ±0.05.

7.1.7.4   SPLP Extraction Fluid #2 – Use this solution with samples from
west of the Mississippi River. Add sufficient 60/40
Sulfuric/Nitric acid solution to reagent water until the pH is
5.00 ±0.05.

7.1.7.5   SPLP Extraction Fluid #3 – This fluid is reagent water and is
used to determine cyanide leachability.

7.1.8   Standard Buffers for pH meter calibration. At a minimum, two
standard buffer solutions are required to bracket the expected pH of
the samples. The solutions shall be separated by at least three pH
units.

8.0   SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1   Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. All soil/sediment samples must be iced or refrigerated at ≤6°C, but not frozen, from the time of collection until receipt at the laboratory.

8.2   Sample Storage

All aqueous/water and soil/sediment samples must be stored at ≤6°C, but not frozen, from the time of sample receipt until preparation.

8.2.1   Unused Sample Storage

Following preparation for percent solids determination or sample characterization, the remaining unused portion of aqueous/water and soil/sediment samples must be returned to storage at a temperature of ≤6°C, but not frozen. After all applicable leaching procedures and/or percent solid determination have been completed, the remaining unused portion of the aqueous/water and soil/sediment samples must be stored within the laboratory until 60 days after delivery of a complete, reconciled data package to the U.S. Environmental Protection Agency (EPA). The Contractor may store these samples at room temperature.

8.2.2   Leachate Sample Storage

The remaining unused portion of the preserved TCLP or SPLP leachates must be stored within the laboratory until 180 days after delivery of a complete, reconciled data package to the EPA. The Contractor may store these samples at room temperature.

8.2.3   Container Storage

The Contractor shall retain the empty sample containers for 60 days after delivery of a complete, reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.3   Contract Required Holding Time

The holding time for TCLP or SPLP extraction for metals samples is 180 days from Validated Time of Sample Receipt (VTSR). The holding time for TCLP or SPLP extraction for mercury samples is 26 days from VTSR. The holding time for SPLP extraction for cyanide samples is 12 days from VTSR.

9.0   CALIBRATION AND STANDARDIZATION

9.1   pH Meter Calibration

Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Each instrument and electrode shall be calibrated at a minimum of two points that bracket the expected pH of the samples. These two points shall be separated by at least three pH units.

Adjust the meter until the readings are within ±0.05 pH units of the buffer solution value.

10.0 PROCEDURE

10.1 Sample Characterization

10.1.1 Percent Solids Determination

Percent Solids determination is based on Standard Method (SM) 2540G, approved 1997.

10.1.1.1 Transfer 5-10 g of sample to a tared weighing boat and record the total weight to the nearest 0.01 g. Sample handling and drying should be conducted in a well-ventilated area.

10.1.1.2 Dry the sample in an oven maintained at 105°C (±5°C) for at least 12 hours, but no more than 24 hours. At the start of drying and at the end of drying, record the oven temperature and date/time.

10.1.1.3 Remove the sample from the oven and allow it to cool in a desiccator.

10.1.1.4 Weigh the sample to the nearest 0.01 g and calculate the percent solids using Equation 1. This value will be used for calculating analytical concentration on a dry weight basis.

EQ. 1 Percent Solids

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

10.1.1.5 Samples containing percent solids less than or equal to 30% shall be prepared at higher sample weights for all analytical methods to yield a dry weight equivalent to the weight range specified in the analytical preparation method.

NOTE: This requirement does not apply to 7-day turnaround or Preliminary Results samples.

10.1.1.5.1 Calculate the required sample weight by dividing the minimal method weight specified in the method by the percent solids expressed as a decimal using Equation 2.

EQ. 2 Required Sample Weight

$$\text{Required Weight} = \frac{\text{Minimal Method Weight}}{\% \text{Solids}/100}$$

10.1.1.6 For samples containing more than 30% solids and less than 50% solids, the Contractor shall proceed with sample analysis and document the issue in the SDG Narrative.

10.1.1.7 For 14 and 21-day turnaround samples without Preliminary Results, the Contractor is required to perform the percent solids determination prior to sample preparation and analysis.

10.1.1.8 Duplicate analyses are not required for percent solids determination.

10.1.2 pH Determinations

10.1.2.1 Aqueous/Water pH Determination

The determination of pH is required for all aqueous/water samples at the time of the receipt at the laboratory. For samples scheduled for Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES), Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), or Mercury analysis, if the pH is >2, the Contractor shall add sufficient nitric acid to the sample to

reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative. For samples scheduled for Cyanide analysis, if the pH is <10, the Contractor shall immediately notify the Sample Management Office (SMO) of the affected sample(s) and pH value(s). SMO will contact the EPA Region. The EPA Region may require the Contractor to either proceed with the analysis or to not analyze the sample(s). The EPA resolution shall be documented in the SDG Narrative.

The Contractor shall follow the procedures for pH measurement based on the EPA SW-846 Method 9041A, Revision 1, July 1992 (pH paper) or the EPA SW-846 Method 9040C, Revision 3, November 2004 [electrometric method (i.e., pH meter and electronic hand-held pen)].

10.1.2.1.1   pH Measurement by pH Paper

Place one or two drops of sample on the pH paper and record the pH for the sample.

10.1.2.1.2   pH Measurement by Electrometric Method

10.1.2.1.2.1   Transfer a sufficient volume of sample to a beaker to cover the sensing elements of the electrode(s) and to give adequate clearance for the magnetic stirring bar. The sample shall not be diluted.

10.1.2.1.2.2   If the sample temperature differs by more than 2°C from the temperature of the buffer solutions used to standardize the meter, the measured pH values must be corrected.

10.1.2.1.2.3   After rinsing and gently wiping the electrode(s) if necessary, immerse the electrode(s) in the sample beaker and stir at a constant rate to provide homogeneity and suspension of solids. The rate of stirring should minimize the air transfer rate at the air/water interface. Record the sample pH and the temperature. Repeat measurements on successive volumes of sample until values differ by less than 0.1 pH units.

10.1.2.2   Soil/Sediment pH Determination

The determination of pH for soil/sediment samples is not required as a routine procedure to be completed at the laboratory. However, if requested at the time of scheduling, the Contractor shall follow the procedures based on the EPA SW-846 Method 9045D, Revision 4, November 2004 to determine the pH by electrometric method (i.e., pH meter or electronic hand-held pen).

10.1.2.2.1   Transfer 20 g of well-mixed sample to a 50 mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 1 hour. Additional water may be added if the soils are hygroscopic or contain large amounts of salts.

10.1.2.2.2   Let the soil suspension stand for at least 1 hour to allow most of the suspended clays to settle. Difficult samples may be filtered or centrifuged to separate the aqueous layer for pH determination. If the supernatant is biphasic, decant the oily phase and measure the pH of the aqueous phase.

10.1.2.2.3   Measure and record the pH for the sample.

10.1.2.2.4   Measure and record the temperature for the sample. If the sample temperature differs by more than 2°C from the temperature of the buffer solutions used to standardize the meter, the measured pH values must be corrected.

10.2   TCLP and SPLP Extraction Procedures

Extraction methods are based on EPA SW-846 Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), Revision 0, July 1992 or EPA SW-846 Method 1312, Synthetic Precipitation Leaching Procedure (SPLP), Revision 0, September 1994.

TCLP vessel and devices must be free of contaminants and cleaned between TCLP samples. Testing procedures shall be performed to ensure the apparatus is functioning properly before proceeding with the extraction.

10.2.1   Preliminary Evaluation

Perform preliminary evaluation on a minimum 100 g sample aliquot. This aliquot will not undergo extraction. These preliminary evaluations include: (1) determination of percent solids by pressure filtration; (2) determination of whether the sample contains insignificant (<0.5%) solids and is therefore its own extract after filtration; (3) determination of whether the solid portion of the sample requires particle size reduction; and for TCLP samples, (4) determination of the appropriate extraction fluid.

10.2.1.1   Preliminary determination of percent solids – For these samples, percent solids is defined as that fraction of a sample (as a percentage of the total sample) from which no liquid can be forced out by applied pressure.

10.2.1.1.1   If a sample will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids), proceed to extraction.

10.2.1.1.2   If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required.

10.2.1.1.2.1   Pre-weigh the filter and the container that will receive the filtrate.

10.2.1.1.2.2   Assemble the filter holder and filter per the manufacturer's instructions. Place the filter on the support screen and secure.

10.2.1.1.2.3   Weigh out at least 100 g of the sample and record the weight.

10.2.1.1.2.4   Allow slurries to stand to permit the solid phase to settle. Samples that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered, followed by filtration of the solid portion of the sample through the same filtration system.

10.2.1.1.2.5   Quantitatively transfer the sample to the filter holder (both liquid and solid phases). Spread the sample evenly over the surface of the filter. If filtration of the waste at a temperature of ≤6°C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm to room temperature in the device before filtering. If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section

10.2.1.1.2.8 to determine the weight of sample that will be filtered.

10.2.1.1.2.6   Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2-minute period), stop the filtration. Note that instantaneous application of high pressure can damage the filter and may cause premature plugging.

10.2.1.1.2.7   The material retained on the filter is defined as the solid phase of the sample, and the filtrate is defined as the liquid phase. Note that certain oily wastes and paint wastes will contain material that appears to be a liquid. However, this material may not filter under pressure filtration. In this case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

10.2.1.1.2.8   Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the sample. Record the weights of the liquid and solid phases. Calculate the percent solids using the following equation:

EQ. 3 Extraction Percent Solids

$$\% \text{ Solids} = \frac{\text{Weight of solid}}{\text{Total Weight of Sample}} \times 100$$

10.2.1.1.2.9   If the percent solids determined in Equation 3 is equal to or greater than 0.5%, then proceed to Section 10.2.1.3 to determine whether the solid material requires particle size reduction.

10.2.1.1.2.10   If it is noticed that a small amount of the filtrate is entrained in wetting of the filter, remove the solid phase and filter from the filtration apparatus. Dry the filter and solid phase at 100°C (±20°C) until two successive weighings yield the same value (within ±1%) and record the weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

10.2.1.1.2.11   Calculate the Percent Dry Solids using the following equation:

EQ. 4 Percent Dry Solids

$$\text{Percent Dry Solids} = \frac{(\text{Wt. of dry waste and filter}) - \text{Tared wt. of filter}}{\text{Initial wt. of waste}} \times 100$$

10.2.1.2   If the percent dry solids is less than 0.5%, then treat the filtrate as the extract. Store this extract at ≤6°C until digestion/distillation.

10.2.1.3   To determine if particle size reduction is required, using a fresh portion of sample, examine the solid portion for particle size. If the material is less than 1 centimeter (cm) in its narrowest dimension (i.e., is capable of passing through a 9.5 mm standard sieve), no particle size reduction is required. Otherwise, prepare the solid portion for extraction by crushing, cutting, or grinding the sample to meet the above criterion.

10.2.1.4   For samples with percent solids greater than 0.5%, determine the appropriate extraction fluid as follows:

10.2.1.4.1   For samples scheduled for TCLP extraction, remove a small aliquot of the sample and reduce the particle size to less than 1 mm. Transfer 5 g of this material to a 500 mL beaker or Erlenmeyer flask.

10.2.1.4.1.1   Add 96.5 mL of reagent water, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use TCLP Extraction Fluid #1 (Section 7.1.7.1). If the pH is ≥5.0, add 3.5 mL 1N HCl (Section 7.1.2), slurry briefly, cover with the watchglass, and heat to 50°C for 10 minutes.

10.2.1.4.1.2   Let the solution cool to room temperature and measure the pH. If the pH is <5.0, use TCLP Extraction Fluid #1 (Section 7.1.7.1); otherwise use TCLP Extraction Fluid #2 (Section 7.1.7.2).

10.2.1.4.2   Use the SPLP extraction fluid appropriate to the information provided on the scheduling document.

10.2.1.4.2.1   For soil samples from east of the Mississippi River, use SPLP Extraction Fluid #1. For samples west of the Mississippi River, use SPLP Extraction Fluid #2.

10.2.1.4.2.2   For cyanide-containing soil/sediments, use SPLP Extraction Fluid #3 (reagent water) because leaching of cyanide-containing samples under acidic conditions may result in the formation of hydrogen cyanide gas and loss of analyte. Along with being potentially hazardous, this results in loss of analyte and makes results useless.

10.2.2   TCLP Sample Extraction

10.2.2.1   A minimum sample size of 100 g is required; however, enough solids shall be extracted to yield a sufficient volume of extract to support all required analyses. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample, and whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid. See Section 10.2.2.3 to determine the approximate amount of extract that will be generated for a given mass with the percent solids determined in Section 10.2.1.1.2.11.

10.2.2.1.1   If the sample is 100% solids, then weigh out 100 g of sample and proceed to Section 10.2.2.3.

10.2.2.1.2   If the sample is less than 0.5% solids, filter enough sample to yield a sufficient volume of extract to support all required analyses if the preliminary percent solids determination did not yield sufficient volume.

10.2.2.1.3   For multiphasic samples with percent solids greater than or equal to 0.5%, but less than 100%, weigh out enough sample to generate a sufficient volume of extract to support all required analyses. Filter the sample using the procedure described in Section 10.2.1. Store the filtrate at ≤6°C, but not frozen.

10.2.2.2   Prepare the solid portion of the sample for extraction by reducing the particle size as described in Section 10.2.1.3. Quantitatively transfer the material into an extractor bottle and include the filter used to separate the initial liquid from the solid phase.

10.2.2.3   Determine the amount of extraction fluid to add to the extractor bottle using the following equation:

EQ. 5 Weight of Extraction Fluid

$$\text{Weight of Extraction Fluid} = \frac{20 \times \text{percent solids} \times \text{Weight of sample filtered}}{100}$$

10.2.2.4   Add this amount of the appropriate extraction fluid (Section 10.2.1.4) to the extractor bottle. Close the bottle tightly (Teflon tape may be used to ensure a tight seal) and secure it in the rotary agitation apparatus. Rotate the samples at 30 rpm (±2 rpm) for 18 hours (±2 hours). Maintain a temperature of 23°C (±2°C) in room where extraction is performed.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of samples (e.g., limed or calcium carbonate-containing sample may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

10.2.2.4.1   Following the 18-hour extraction, separate the material in the extractor bottle into its component liquid and solid phase by filtering through a new glass filter as described in Section 10.2.1.1. For the final filtration of the extract, the glass fiber filter may be changed as necessary during filtration.

10.2.2.4.2   If the sample was 100% solids, this filtered liquid is the extract.

10.2.2.4.3   For multiphasic samples, combine this extract with the filtrate generated in Section 10.2.2.1.3 if the two liquids are miscible. If the two liquids are not miscible, they shall be prepared and analyzed separately and the analytical results mathematically combined.

10.2.2.4.4   Record the pH of the final extract. Preserve extracts for metals (both ICP-AES and Hg) with nitric acid to pH <2. Preserve SPLP extracts for Cyanide analysis with NaOH to pH >10 and store at ≤6°C.

10.2.3   SPLP Sample Extraction

> The Contractor shall follow the procedures in Section 10.2.2 using the appropriate extraction fluid specified in Section 10.2.1.4.2.

11.0   DATA ANALYSIS AND CALCULATIONS

> See individual procedures in Section 11.0 for data analysis and calculations.

12.0   QUALITY CONTROL

12.1   Leachate Extraction Blank

12.1.1   The Leachate Extraction Blank (LEB) shall contain all the reagents and in the same volumes as used in extracting the samples. The LEB shall be carried through the complete extraction procedure.

12.1.2   At least one LEB, consisting of reagent water processed through the extraction procedure, shall be extracted with every SDG scheduled for TCLP or SPLP.

12.1.3   Each Complete SDG File (CSF) shall contain the results of all LEB analyses associated with the samples in that SDG.

12.1.4   The LEB(s) result(s) is (are) to be reported for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination.

12.1.5   Under no circumstances should the LEB be analyzed at a dilution.

12.2   Summary of Quality Control Operations

> The Quality Control (QC) operations performed are summarized Section 17.0, Table 1 – Quality Control Operations.

13.0   METHOD PERFORMANCE

> Not applicable.

14.0   POLLUTION PREVENTION

> See Section 13.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

15.0   WASTE MANAGEMENT

> See Section 14.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

16.0   REFERENCES

16.1   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1311, Revision 0, Update III, July 1992.

16.2   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 1312, Revision 0, Update III, September 1994.

16.3   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 9040C, Revision 3, November 2004.

16.4   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid
       Waste, Physical/Chemical Methods (SW-846), Method 9041A, Revision 1,
       July 1992.

16.5   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid
       Waste, Physical/Chemical Methods (SW-846), Method 9045D, Revision 4,
       November 2004.

17.0   TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. QUALITY CONTROL OPERATIONS

QC Operation	Frequency
Leachate Extraction Blank (LEB)	For each SDG, an LEB for each extraction procedure.

EXHIBIT D

INDUCTIVELY COUPLED PLASMA -
ATOMIC EMISSION SPECTROSCOPY

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Exhibit D – Analytical Methods for Inductively Coupled Plasma – Atomic
Emission Spectroscopy

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Exhibit D – Analytical Methods for Inductively Coupled Plasma – Atomic
Emission Spectroscopy

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1.0   SCOPE AND APPLICATION

This exhibit provides procedures for the use of Inductively Coupled
Plasma - Atomic Emission Spectroscopy (ICP-AES) to determine the
concentration of total recoverable and dissolved metals in
aqueous/water, leachate derived from the Toxicity Characteristic
Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure
(SPLP), soil/sediment, and wipe samples taken from hazardous waste
sites. All metals contained in the Inorganic Target Analyte List (TAL)
for ICP-AES in Exhibit C - Inorganic Target Analyte List and Contract
Required Quantitation Limits are quantitated by this method.

2.0   SUMMARY OF METHOD

2.1   General Method Overview

This method describes the multi-element determination of trace metals
by ICP-AES. Aqueous/water, TCLP/SPLP leachate, soil/sediment, and wipe
samples are treated with acids and heat to solubilize the metals
present. These digestates are then analyzed for trace metals by an
atomic emission optical spectroscopic technique. Samples are nebulized
and the aerosol that is produced is transported to a plasma torch where
excitation occurs. Characteristic atomic-line emission spectra are
produced by a radio-frequency inductively coupled plasma. The spectra
are dispersed and the intensities of the lines are monitored by a
photosensitive device. The signals from the photosensitive device are
processed by a computer. A background correction technique is required
to compensate for variable background contribution to the spectra of
trace elements. Background must be measured adjacent to analyte lines
on samples during analysis. The position selected for the background
intensity measurement, on either or both sides of the analytical line,
will be determined by the complexity of the spectrum adjacent to the
analyte line. The position used must be free of spectral interference
and reflect the same change in background intensity as occurs at the
analyte wavelength measured. Background correction is not required in
cases of line broadening where a background correction measurement
would actually degrade the analytical result.

2.2   Summary of Digestion Procedures

2.2.1   Hotplate Acid Digestion of Aqueous/Water and TCLP/SPLP Leachate
        Samples (based on EPA Method 200.7)

2.2.2   Hotplate Acid Digestion of Soil/Sediment Samples (based on EPA
        Method 3050B)

2.2.3   Hotplate Acid Digestion of Wipe Samples (based on EPA Method 3050B)

3.0   DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0   INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements in aqueous/waters, TCLP/SPLP leachates, soil/sediments, and wipes by ICP-AES. To prevent this, appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 milligrams/Liter (mg/L) and when total elements are determined after the appropriate digestion procedures are performed. Several types of interferences are given in Sections 4.1 through 4.3 below.

4.1   Spectral Interferences

Spectral interferences can be categorized as: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; (4) and/or background contribution from stray light from the line emission of high concentration elements. The first effect can be compensated by utilizing a computer correction of the raw data. This would require the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array.

4.2   Physical Interferences

Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies, especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may minimize these interferences. If these types of interferences are present, they must be reduced by dilution of the sample.

Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution has been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

Internal standardization may be effectively used to compensate for many physical interference effects.

4.3   Chemical Interferences

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with ICP-AES technique; however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, and by matrix matching. These types of interferences can be highly dependent on matrix type and the specific element.

5.0   SAFETY

See Section 12.0 in Exhibit D - Introduction to Inorganic Analytical
Methods.

6.0   EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes
only. No endorsement is implied. Equivalent performance may be
achieved using equipment and supplies other than those specified here;
however, a demonstration of equivalent performance meeting the
requirements of this Statement of Work (SOW) is the responsibility of
the Contractor. The Contractor shall document any use of alternate
equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1   Glassware/Labware

6.1.1   250 milliliter (mL) beaker or other appropriate digestion vessel
(glass or plastic)

6.1.2   Watch glasses (glass or plastic)

6.1.3   Funnels

6.1.4   Graduated cylinders

6.1.5   Various volumetric flasks (Class A) and calibrated pipettes.
Manufacturer's instructions should be followed for the calibration
and maintenance of adjustable pipettes.

6.1.6   Thermometer that covers a range of 0-200°C

6.1.7   Whatman No. 42 filter paper (or equivalent)

6.1.8   Hotplate, block digester, or other heating source capable of
maintaining 95°C (±3°C)

6.1.9   Balances – Top loader balance, 300 gram (g) capacity, and a minimum
sensitivity of ±1.0 mg

A balance calibration must be checked with known masses once per
each day of use. This verification consists of a check with two
weights covering the range expected (approximately ±50% of the
expected measured mass) for each balance and be accurate to ±1.0 mg.
The masses that are used to check the balances daily must be checked
on a monthly basis using National Institute of Standards and
Technology (NIST)-traceable known reference masses (Class '1' or
'2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier
Class 'S' defined masses). All balances must be checked at least
once annually by a certified technician. The reference masses used
by the Contractor must be recertified every five years or sooner if
there is reason to believe damage (corrosion, nicks) has occurred.
The Contractor shall maintain documentation that demonstrates that
these criteria have been met.

6.2   Inductively Coupled Plasma - Atomic Emission Spectrometer

The ICP-AES consists of:

- A computer-controlled atomic emission spectrometer with background
  correction;

- A radio-frequency generator; and

- A supply of Argon gas, welding grade or better.

7.0   REAGENTS AND STANDARDS

7.1   Reagents

7.1.1   Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions.

7.1.2   Hydrochloric acid - Concentrated 32-38% (specific gravity 1.19).

7.1.3   Hydrochloric acid (50% v/v) - Add 500 mL of conc. hydrochloric acid to 400 mL reagent water and dilute to 1 L.

7.1.4   Nitric acid - Concentrated 67-70% (specific gravity 1.41).

7.1.5   Nitric acid (50% v/v) - Add 500 mL conc. nitric acid to 400 mL reagent water and dilute to 1 L.

7.1.6   Hydrogen peroxide (30%).

7.1.7   Nitric acid (2% v/v) - Add 20 mL conc. nitric acid to 500 mL reagent water and dilute to 1 L.

7.2   Standards

7.2.1   Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit D - Introduction to Inorganic Analytical Methods, Section 11.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Samples, sample digestates, and standards must be stored separately.

7.2.2   Stock Standard Solutions

Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals (at least 99.99% pure). All salts must be dried for 1 hour at 105°C unless otherwise specified.

CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.

7.2.3   Secondary Dilution Standards

Prepare mixed secondary dilution standard solutions by diluting the appropriate volumes of stock standards with 2% (v/v) nitric acid, or as recommended by the manufacturer, to obtain the final volume. Mixed secondary dilution standard solutions may be purchased. The purchased standards shall meet the requirements in Section 7.2.1.

7.2.4   Working Standards

7.2.4.1   Interference Check Sample Solution

7.2.4.1.1   The Interference Check Sample (ICS) consists of two solutions: ICS Solution A (ICSA) and ICS Solution AB (ICSAB). ICSA consists of the interferents and ICSAB consists of the analytes mixed with the interferents. The ICS standards (ICSA for the interferents only and ICSB for the analytes only) shall be obtained from the EPA.

7.2.4.1.1.1   Only if the ICS solutions are not available from the EPA, ICSs shall be prepared with interferent and analyte concentrations at the levels specified in Table 1 – Interferent and Analyte Concentrations Used for ICP-AES Interference Check Sample (ICS).

7.2.4.1.1.2   If the solutions are prepared, the mean value and standard deviation shall be established by initially analyzing the ICSs at least five times repetitively for each analyte listed. Results shall be within the control limits of ±20% of the established mean value or ±1 times the analyte's Contract Required Quantitation Limit (CRQL) of the established mean value, whichever is greater. The mean and standard deviation shall be reported in the raw data.

7.2.4.2   Mixed Calibration Standard Solutions

Care should be taken when preparing the mixed standards that the analytes are compatible and stable. Fresh mixed standards should be prepared as needed with the realization that concentration can change with aging.

Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interferences or the presence of impurities. Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add 2 mL of nitric acid and dilute to 100 mL with reagent water or these standards can be matrix matched to the digested samples. The analyte concentrations in the calibration standards should be sufficient to produce good measurement precision and to accurately define the slope of the response curve. Transfer the mixed standard solutions to Fluorinated Ethylene Propylene (FEP) fluorocarbon or unused polyethylene bottles for storage.

7.2.4.3   Initial Calibration Verification Solutions

7.2.4.3.1   The Initial Calibration Verification (ICV) solution(s) shall be obtained from the EPA.

7.2.4.3.1.1   If the solution(s) is (are) not available from the EPA, then the ICV solution shall be prepared by the laboratory using a certified solution for each analyte from an independent source. An independent source is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range.

7.2.4.3.1.2   The ICV standard shall be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier.

7.3   Blanks

Three types of blanks are required for this method. A Calibration Blank is used to establish the analytical calibration curve, a Preparation Blank (see Section 12.1) is used to assess possible contamination from the sample preparation procedure and to assess spectral background, and a rinse solution is used to flush the instrument between samples to reduce memory interferences.

7.3.1 Calibration Blank - Consists of 2% (v/v) nitric acid in reagent water or matrix matched to the digested samples. The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are identical in composition to the Calibration Blank and are used to monitor for analyte carryover.

7.3.2 Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Preparation Blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Soil/sediment blanks shall use 1.00 mL (±0.01 mL) of reagent water.

7.3.3 Rinse Solution – Must contain sufficient nitric acid to allow the instrument to return to baseline between the analysis of digested samples, blanks, and standards. The rinse solution would typically consist of 2% (v/v) nitric acid in reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Wipe samples may be placed in zip-top plastic bags, glass, or polyethylene wide-mouth containers for shipment. Wipe samples are not preserved. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All soil/sediment samples must be iced or refrigerated at ≤6°C but not frozen from the time of collection until receipt at the laboratory.

8.2 Sample Storage

All aqueous/water and soil/sediment samples must be stored at ≤6°C but not frozen from the time of sample receipt until digestion. Wipe samples shall remain in their original bags until preparation and may be stored at room temperature within the laboratory.

8.2.1 Unused Sample Storage

The Contractor shall retain the unused portion of aqueous/water and soil/sediment samples for a period of 60 days after delivery of a complete, reconciled data package to the EPA. The samples may be stored at room temperature.

8.2.2 Digestate Sample Storage

Sample digestates must be stored until 180 days after delivery of a complete, reconciled data package to the EPA. The Contractor shall store sample ICP-AES digestates in plastic bottles. The bottles shall be labeled with the EPA Sample Number, Case Number, SDG Number, MA No. (if applicable), and digestion date. A logbook of stored digestates, listing the EPA Sample Numbers and associated Case and SDG Numbers, shall be maintained.

8.2.3 Container Storage

The Contractor shall retain the empty sample containers for 60 days after the delivery of a complete, reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.3   Contract Required Holding Time

The holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of TCLP or SPLP leachates is 180 days from the date of extraction.

9.0   CALIBRATION AND STANDARDIZATION

9.1   Instrument Operating Parameters

Because of the differences between various manufacturers and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL), precision, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements used to determine interelement corrections must be within the instrument operating range. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2   Pre-Calibration Routine

Prior to calibration, the Contractor shall set up each instrument with the operating parameters established in Section 9.1 and conduct any necessary adjustments to the plasma conditions. The instrument must be allowed to become stable (usually 30 minutes) before calibration is performed.

9.3   Instrument Calibration Procedure

9.3.1   Summary of Instrument Calibration

Prior to sample analysis, the Contractor shall calibrate each instrument to determine sensitivity and linearity of the response.

9.3.2   Frequency of Instrument Calibration

Each instrument shall be calibrated each time it is turned on, set up, or after ICV, ICB, Continuing Calibration Verification (CCV), or CCB failure. The instrument calibration date and time shall be included in the raw data.

9.3.3   Procedure for Instrument Calibration

9.3.3.1   Each instrument shall be calibrated according to the manufacturer's recommended procedures.

9.3.3.2   At least six calibration standards shall be used for each analyte. The calibration standards shall be prepared as in Section 7.2.4.2. One of the standards shall be a blank standard and one shall be at or below the CRQL but greater than the MDL. The rest of the standards shall be uniformly spread out in graduated amounts over the appropriate calibration range of the analyte.

9.3.3.3   A minimum of three replicate integrations are required for data acquisition. The Contractor shall use the average of all the integrations for instrument calibration and data reporting.

9.3.4   Calculations for Instrument Calibration

9.3.4.1   The calibration curve shall be calculated for each analyte using linear regression by plotting the concentration of the standard [in micrograms/Liter (µg/L)] on the X-axis versus the corrected instrument response on the Y-axis. The corrected instrument responses are those corrections [e.g., correction for background, Interelement Corrections (IECs), calibration blank] that may be applied to the raw uncorrected instrument response prior to determining the calibration curve.

9.3.4.2   The Contractor may use standard linear regression, weighted linear regression (1/concentration, 1/square of concentration, variance, 1/variance, standard deviation, 1/standard deviation, or 1/square of standard deviation), or linear regression with zero force calibration models, as appropriate, for the above calculation. No other types of equations (e.g., quadratic) are to be used.

9.3.4.3   The calibration curve for each analyte shall be checked to establish the representativeness of the data that were used to produce it. This check involves re-fitting the data of the non-blank calibration standards back to the calibration curve, and computing the difference between the calculated concentration and the expected concentration for each of these standards. The difference is then divided by the expected concentration of the respective standard and multiplied by 100.

9.3.5   Technical Acceptance Criteria for Instrument Calibration

9.3.5.1   The correlation coefficient of the calibration curve shall be greater than or equal to 0.995.

9.3.5.2   The Percent Difference (%D) for each of the standards shall be within the control limits of ±30%.

9.3.5.3   If a standard is analyzed for a particular analyte at a concentration that is below the CRQL and the %D criteria is not met, that standard can be excluded from the calibration curve as long as the lowest non-zero standard for that analyte is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.3.6   Corrective Action for Instrument Calibration

9.3.6.1   Sample analysis shall not begin until the criteria described in Section 9.3.5 have been met.

9.3.6.2   Any changes or corrections to the analytical system shall be followed by recalibration.

9.4   Initial Calibration Verification

9.4.1   Summary of Initial Calibration Verification

Prior to sample analysis, the Contractor shall demonstrate the accuracy of the instrument calibration for each instrument through the analysis of an ICV standard.

9.4.2   Frequency of Initial Calibration Verification

The ICV shall be analyzed immediately after the instrument has been calibrated.

9.4.3   Procedure for Initial Calibration Verification

9.4.3.1   The ICV shall be analyzed at each wavelength used to report final results for each analyte.

9.4.3.2   The ICV shall reflect the conditions of analysis of the associated analytical samples.

9.4.4   Calculations for Initial Calibration Verification

9.4.4.1   The Percent Recovery (%R) of the ICV shall be calculated using the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{Found(ICV)}{True(ICV)} \times 100$$

WHERE,
Found (ICV)   = The found concentration of the analyte in the ICV Solution
True (ICV)   = The expected concentration of the analyte in the ICV Solution

9.4.4.2   The Percent Relative Standard Deviation (%RSD) from all replicate integrations shall be calculated for each wavelength used to report final results using Equations 2, 3, and 4.

EQ. 2 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

WHERE,
SD   = Standard deviation of ICV replicates (per analyte) from EQ. 3
$\overline{X}$ = Mean value of the ICV replicates (per analyte) from EQ. 4

9.4.4.3   Equation 3 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 3 Standard Deviation Calculation

$$SD = \sqrt{\frac{\sum\limits_{i=1}^{n}(X_i - \overline{X})^2}{(n-1)}}$$

WHERE,
X_i = Each individual value used to calculate the mean
$\overline{X}$ = The mean of n values from EQ. 4
n   = Total number of values

9.4.4.4   Equation 4 is the general formula for the mean of a set of values ($\overline{X}$).

EQ. 4 Mean Value Calculation

$$\overline{X} = \frac{\sum\limits_{i=1}^{n} X_i}{n}$$

WHERE,

X_i = Each individual value used to calculate the mean

n = Total number of values

9.4.5   Technical Acceptance Criteria for Initial Calibration Verification

9.4.5.1   The ICV %R shall be within the control limits of 90-110%.

9.4.5.2   The %RSD of the ICV integrations shall be less than or equal to 5.0%.

9.4.6   Corrective Action for Initial Calibration Verification

If the recovery is outside the control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

9.5   Continuing Calibration Verification

9.5.1   Summary of Continuing Calibration Verification

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration through the analysis of a CCV standard. The Contractor shall not analyze a blank immediately prior to the analysis of the CCV.

9.5.2   Frequency of Continuing Calibration Verification

9.5.2.1   A CCV shall be analyzed at the beginning of the analytical sequence and after the last analytical sample. A CCV shall also be analyzed at a frequency not to exceed every 2 hours during an analytical sequence. See the example analytical sequence in Section 10.4.5.

9.5.2.2   The analytical sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCV standards meet the technical acceptance criteria in Section 9.5.5.

9.5.3   Procedure for Continuing Calibration Verification

9.5.3.1   The CCV standard shall be prepared using the same source and in the same acid matrix as the calibration standards by combining compatible analytes at a concentration at or near the mid-level of their respective calibration curve.

9.5.3.2   The same CCV standard shall be used throughout the analytical sequences for an SDG.

9.5.3.3   The CCV shall be analyzed at each wavelength used to report final results for each analyte.

9.5.3.4   Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5.4   Calculations for Continuing Calibration Verification

9.5.4.1   The %R of the CCV shall be calculated using the following equation:

EQ. 5 CCV Percent Recovery

$$\%R = \frac{Found\ (CCV)}{True(CCV)} \times 100$$

WHERE,

Found (ICV) = The found concentration of the analyte in the CCV Solution

True (ICV) = The expected concentration of the analyte in the CCV Solution

9.5.4.2 The %RSD from all replicate integrations shall be calculated for each wavelength used to report final results using Equations 2, 3, and 4 above.

9.5.5 Technical Acceptance Criteria for Continuing Calibration Verification

9.5.5.1 The CCV %R shall be within the control limits of 90-110%.

9.5.5.2 The %RSD of the CCV integrations shall be less than or equal to 5.0%.

9.5.5.3 All samples shall be analyzed within 2 hours of an acceptable opening and closing CCV.

9.5.6 Corrective Action for Continuing Calibration Verification

If the deviations of the CCV are greater than the specified control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected.

9.6 Initial and Continuing Calibration Blank

9.6.1 Summary of Calibration Blank

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration baseline through the analysis of an ICB and CCB.

9.6.2 Frequency of Calibration Blank

9.6.2.1 An ICB shall be analyzed immediately after the instrument has been calibrated and the calibration verified by the analysis of the ICV.

9.6.2.2 Each CCB shall be analyzed immediately after the analysis of a CCV.

9.6.3 Procedure for Calibration Blank

9.6.3.1 The ICB and CCB samples shall be analyzed at each wavelength used for reporting final results for each analyte.

9.6.3.2 The ICB analyzed shall reflect the conditions of analysis of the associated analytical samples.

9.6.3.3 Each CCB analyzed shall reflect the conditions of analysis of the associated analytical samples (the preceding analytical samples up to the previous CCB as applicable and the analytical samples following this CCB up to the next CCB as applicable).

9.6.4   Calculations for Calibration Blank

The results for the ICB and CCB samples shall be calculated using Equation 6 in Section 11.0.

9.6.5   Technical Acceptance Criteria for Calibration Blank

The absolute value of each calibration blank result shall be less than or equal to the CRQL for aqueous/water samples for the analyte.

9.6.6   Corrective Action for Calibration Blank

If the absolute value of the calibration blank exceeds the CRQL for aqueous/water samples, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all affected analytical samples analyzed since the last compliant calibration blank performed for the analytes affected.

10.0   PROCEDURE

10.1   Aqueous/Water Sample Preparation

Preparation Method 200.7 [based on EPA NERL Method 200.7, Revision 4.4 (1994)]

10.1.1   If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.

10.1.2   For the determination of total recoverable analytes in aqueous/water and leachate samples, transfer a 100 mL (±1 mL) aliquot from a well-mixed, acid-preserved sample to an appropriately sized (approximately 250 mL) digestion vessel (e.g., a beaker or hot block digestion tube). The sample shall not be diluted prior to digestion.

NOTE: A reduced sample volume of 50 mL can be used. If this reduced volume is used, then all other reagents and volumes shall be reduced appropriately.

10.1.3   Add 2 mL 50% (v/v) nitric acid and 1 mL 50% (v/v) hydrochloric acid to the beaker containing the measured volume of sample. Place the beaker on a hotplate, or other comparable heating device, for solution evaporation. The hotplate should be located in a fume hood and previously adjusted to provide evaporation at a temperature of 95°C (±3°C), when covered. The beaker should be covered with an elevated watch glass or other necessary steps should be taken to prevent sample contamination from the fume hood environment.

10.1.4   Reduce the volume of the sample aliquot to about 20 mL by gently heating at 95°C (±3°C). **DO NOT BOIL.** This step takes about 2 hours for a 100 mL aliquot with the rate of evaporation rapidly increasing as the sample volume approaches 20 mL. (A spare beaker containing 20 mL of water can be used as a gauge.)

10.1.5   Cover the beaker with a watch glass to reduce additional evaporation and gently reflux the sample for 30 minutes. (Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the HCl-H_2O azeotrope.)

10.1.6   Allow the beaker to cool. Quantitatively transfer the sample solution to a 100 mL volumetric flask, make to volume with reagent water, stopper, and mix.

10.1.7   Allow any undissolved material to settle overnight, or centrifuge or filter a portion of the prepared sample until clear to avoid plugging the nebulizer with solid particles.

10.1.8   The sample is now ready for analysis. Because the effects of various matrices on the stability of the samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

10.1.8.1   The digested sample may be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves, according to Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0.

10.2   Soil/Sediment Sample Preparation

    Preparation Method 3050B [based on EPA Method 3050B, Revision 2, (December 1996)]

10.2.1   Mix the sample thoroughly to achieve homogeneity. Weigh to the nearest 0.01 g and transfer 1.00 – 1.50 g sample (wet weight) to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube).

10.2.2   Add 10 mL of 50% (v/v) nitric acid, mix the slurry, and cover with a watch glass. Heat the sample to 95°C (± 3°C) and reflux for 10 to 15 minutes without boiling. Allow the sample to cool, add 5 mL of concentrated nitric acid, replace the cover, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by the nitric acid, repeat this step (addition of 5 mL of concentrated nitric acid) until no brown fumes are given off by the sample indicating the complete reaction with nitric acid. Using a watch glass, either allow the solution to evaporate to approximately 5 mL without boiling or heat at 95°C (± 3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.2.3   After the sample has cooled, add 2 mL of reagent water and 3 mL of 30% hydrogen peroxide. Cover the vessel with a watch glass and return the covered vessel to the heat source for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides and cool the vessel. Continue to add 30% hydrogen peroxide in 1-mL amounts with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL of 30% hydrogen peroxide.) Cover the sample with a watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95°C (± 3°C) without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times.

10.2.4 After the sample has cooled, add 10 mL of concentrated hydrochloric acid to the sample digestate and cover with a watch glass. Place the sample on/in the heating source and reflux at 95°C (±3°C) for 15 minutes. Let the sample digestate cool.

10.2.5 Filter the sample digestate through Whatman No. 42 filter paper (or equivalent) and collect the filtrate in a 100-mL volumetric flask. Rinse the filter paper with a small amount of reagent water to complete the quantitative transfer of the analytes and collect the liquid in the same 100-mL volumetric flask. The solution being analyzed must be clear to avoid plugging the nebulizer with solid particles. Make to volume with reagent water, stopper, and mix.

NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

10.2.6 The sample is now ready for analysis.

10.2.6.1 The digested sample may be diluted if high levels of interferences are noted or if any of the analytes exceed the upper limit of their respective calibration curves, according to Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0.

10.3 Wipe Sample Preparation

Preparation Method 3050B [based on EPA Method 3050B, Revision 2 (December 1996)]

10.3.1 Transfer the wipe to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube). If material remains in the original sample container, use a small (5 mL) portion of reagent water to rinse the material into the digestion vessel.

10.3.2 Follow the procedure as described in Sections 10.2.2 through 10.2.6.

10.4 Sample Analysis

10.4.1 It is recommended that a semi-quantitative analysis be conducted to screen for high element concentrations that may be beyond the calibration range of the instrument or high levels of interferences.

10.4.2 For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for data reporting.

10.4.3 In accordance with the instrument manufacturer's instructions, a rinse solution should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample. Samples should be aspirated for a sufficient period of time to obtain a stable response prior to the collection of data.

10.4.4 Sample digestates having high levels of interferences or concentrations higher than the established calibrated range as determined by the expected concentration of the highest calibration standard shall be diluted into range and reanalyzed, according to procedure in Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0.

10.4.5   Example Analytical Sequence for ICP-AES Including the Instrument
         Calibration:

         S##
         S##
         S##
         S##
         S##
         S##
         ICV
         ICB
         ICSA
         ICSAB
         CCV###
         CCB###
         samples
         CCV###
         CCB###
         samples
         CCV###
         CCB###, etc.

11.0 DATA ANALYSIS AND CALCULATIONS

      Calculate the Target Analyte concentration using the following
      equations.

11.1 Aqueous/Water and TCLP/SPLP Leachate Sample Calculation

      EQ. 6 Aqueous/Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Concentration}(\mu g/L) = C \times \frac{V_f}{V} \times DF$$

      WHERE,
      C   =   Instrument value in µg/L (The average of all replicate
               exposures)
      V_f =   Final digestion volume (mL)
      V   =   Initial aliquot amount (mL)
      DF   =   Dilution Factor

      NOTE: Convert units to mg/L for TCLP leachates by dividing the final
            calculated concentration by 1000.

11.2 Soil/Sediment Sample Calculation

      The concentrations determined in the digestate are to be reported on
      the basis of the dry weight of the sample, in units of mg/kg:

      EQ. 7 Soil/Sediment Sample Concentration

$$\text{Concentration (mg/kg dry weight)} = C \times \frac{V_f}{W \times S} \times DF \ / \ 1000$$

      WHERE,
      C   =   Instrument value in µg/L (The average of all replicate
               exposures)
      V_f =   Final digestion volume (mL)
      W   =   Initial aliquot amount (g)
      S   =   % Solids/100 (Exhibit D – General Inorganic Analysis,
               Section 10.1.1)
      DF   =   Dilution Factor

11.3 Wipe Sample Calculation

EQ. 8 Wipe Mass

$$Mass\,(\mu g) = C \times V_f \times DF / 1000$$

WHERE,
C   =   Instrument value in µg/L (The average of all replicate exposures)
V_f =   Final digestion volume (mL)
DF   =   Dilution Factor

11.4 Adjusted Contract Required Quantitation Limit Calculation

11.4.1 Calculate the adjusted CRQL for aqueous/water or TCLP/SPLP leachate samples, by multiplying the CRQL (µg/L) by the sample dilution factor and the V_f/V term as noted in Equation 6. Convert units to mg/L for TCLP leachate samples.

11.4.2 Calculate the adjusted CRQL for soil/sediment samples using the following equation:

EQ. 9 Adjusted Soil/Sediment CRQL

$$Adjusted\ CRQL\ (mg/kg) = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

WHERE,
C   =   CRQL (mg/kg)
W_M =   Minimum method required aliquot amount (g) (1.00 g)
W   =   Initial aliquot amount (g)
V_M =   Method required final sample digestion volume (mL) (100 mL or 50 mL)
V_f =   Final digestion volume (mL)
S   =   % Solids/100 (see Exhibit D – General Inorganic Analysis, Section 10.1.1)
DF   =   Dilution Factor

11.5 Hardness (Total) Sample Calculation

Total Hardness is defined as the sum of calcium and magnesium concentrations, expressed as calcium carbonate in mg/L. Total Hardness is calculated according to Standard Method 2340 B.

EQ. 10 Calculation of Hardness (Total) in Aqueous/Water Samples

Hardness (mg/L) = [Conc. Ca (mg/L) × 2.497]+[Conc. Mg (mg/L) × 4.118]

WHERE,
Conc. Ca (mg/L) = Calcium concentration (µg/L) ÷ 1000
Conc. Mg (mg/L) = Magnesium concentration (µg/L) ÷ 1000

12.0 QUALITY CONTROL

12.1 Preparation Blank Sample

12.1.1   Summary of Preparation Blank Sample

The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.1.2   Frequency of Preparation Blank Sample

12.1.2.1   At least one Preparation Blank shall be prepared with every SDG, or with each preparation batch, whichever is more frequent.

12.1.2.2   If sufficient clean wipes are provided by the sampler, an additional Preparation Blank for the wipe samples shall be prepared using a clean wipe.

12.1.3   Procedure for Preparation Blank Sample

The Preparation Blank shall be carried through the complete sample preparation procedure for the matrix and contain the same acid concentration in the final digestate as the samples.

12.1.4   Calculations for Preparation Blank Sample

Calculate the results for aqueous Preparation Blanks by using Equation 6. Calculate the results for soil/sediment Preparation Blanks by using Equation 7. Calculate the results for wipe Preparation Blanks by using Equation 8.

12.1.5   Technical Acceptance Criteria for Preparation Blank Sample

12.1.5.1   The absolute value of the Preparation Blank result shall be less than or equal to the CRQL.

12.1.5.2   For aqueous/water and soil/sediment samples, any analyte concentration in the Preparation Blank may be greater than the CRQL, if the concentration of the analyte in the associated samples is greater than or equal to 10 times the blank concentration.

12.1.5.3   For aqueous/water and soil sediment samples, any analyte concentration in the Preparation Blank may be less than the negative CRQL, if the concentration in the associated samples is greater than or equal to 10 times the CRQL.

12.1.6   Corrective Action for Preparation Blank Sample

12.1.6.1   For aqueous/water and soil/sediment samples, if any analyte concentration in the Preparation Blank is greater than the CRQL, and the concentration of the analyte in any of the associated samples is less than 10 times the blank concentration, then all samples with less than 10 times the blank concentration shall be reprepared and reanalyzed with appropriate new QC for that analyte.

12.1.6.2   For aqueous/water and soil/sediment samples, if any analyte concentration in the Preparation Blank is less than the negative CRQL, and the concentration in the associated samples is less than 10 times the CRQL, then all samples with less than 10 times the CRQL concentration shall be reprepared and reanalyzed with appropriate new QC for that analyte.

12.1.6.3   If the results of the Preparation Blank for wipes exceed either the CRQL or are less than the negative CRQL, the Contractor shall note this in the SDG Narrative, since wipe samples are fully consumed by initial analysis and cannot be reprepared and reanalyzed.

12.2   Interference Check Sample

12.2.1   Summary of Interference Check Sample

The Contractor shall analyze the ICS to verify interelement and background correction factors.

12.2.2   Frequency of Interference Check Sample

The Contractor shall analyze, quantitate, and report the results for all elements on the TAL and for all interferents (target and non-target) immediately after the initial calibration sequence, but not before the ICV/ICB. The analysis of the ICS shall be immediately followed by the analysis of a CCV/CCB pair.

12.2.3   Procedure for Interference Check Sample

12.2.3.1   The ICS solutions (Section 7.2.4.1) shall be analyzed according to the instructions supplied with the ICS.

12.2.3.2   The Contractor shall initially analyze the ICSA and the ICSAB undiluted. Any dilution of the ICS (for the highest concentration elements) necessary to meet the calibrated range values of the instrument shall be analyzed after the undiluted analyses of the ICSA and ICSAB.

12.2.3.3   An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

12.2.4   Calculations for Interference Check Sample

12.2.4.1   The ICSA/ICSAB sample concentrations shall be calculated using Equation 6.

12.2.4.2   The %R of the ICSA and ICSAB shall be calculated using the following equations:

EQ. 11 ICSA Percent Recovery

$$\%R = \frac{\text{Found (ICSA)}}{\text{True (ICSA)}} \times 100$$

WHERE,
Found (ICSA) =   The found concentration of the analyte in the ICSA Solution
True (ICSA)   =   The expected concentration of the analyte in the ICSA Solution

EQ. 12 ICSAB Percent Recovery

$$\%R = \frac{\text{Found (ICSAB)}}{\text{True (ICSAB)}} \times 100$$

WHERE,
Found (ICSAB) = The found concentration of the analyte in the ICSAB Solution
True (ICSAB)   = The expected concentration of the analyte in the ICSAB Solution

12.2.5   Technical Acceptance Criteria for Interference Check Sample

12.2.5.1   The ICSA and ICSAB %R shall be within the control limits of ±20% of the analyte's true value or the results shall be within ±1 times the CRQL of the analyte's true value, whichever is greater. If the true value for a given analyte is not listed in the certified values for the solution, then the true value shall be assumed to be zero and the ±1 times the CRQL control limits shall apply.

For example, for Chromium (CRQL = 10 µg/L, ICSA true value = 43 µg/L) the correct control window to use would be the greater of ±20% of the true value (0.20 x 43 µg/L = ±8.6 µg/L) or ±1 times the CRQL (±10 µg/L). Therefore, the control window for the found value for Chromium in the ICSA is 43±10, or 33 to 53 µg/L.

12.2.5.2   Only if the ICS solutions are not available from the EPA, the %R of the prepared independent Check Sample results shall be within the control limits of ±20% of the established mean value or the results shall be within ±1 times the analyte's CRQL of the established mean value, whichever is greater.

12.2.6   Corrective Action for Interference Check Sample

If the deviations of the ICSA and/or the ICSAB are greater than the specified control limits, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples. New IECs may also need to be determined for the failed analyte(s). For analytes with CRQLs less than 1000 µg/L, the ICSA and ICSAB results shall be reported from an undiluted sample analysis.

12.3 Matrix Spike and Post-Digestion Spike Samples

12.3.1   Summary of Matrix Spike and Post-Digestion Spike Sample

The Matrix Spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

12.3.2   Frequency of Matrix Spike and Post-Digestion Spike Samples

12.3.2.1   At least one Matrix Spike sample analysis shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[1]

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[1] The EPA may require additional spike sample analyses, upon request from the EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR).

12.3.2.2   Matrix Spike sample analysis is not required for wipe samples.

12.3.2.3   If a Matrix Spike sample does not meet the technical acceptance criteria listed in Section 12.3.5, a Post-Digestion Spike sample shall be performed for those analytes that do not meet the specified criteria (exception: Ag).

12.3.3   Procedure for Matrix Spike and Post-Digestion Spike Samples

12.3.3.1   For a Matrix Spike sample, the spike is added before the digestion (i.e., prior to the addition of other reagents).

12.3.3.2   The analyte spike shall be added in the amount given in Table 2 – Spiking Levels for Matrix Spike Sample Analyses, for each element analyzed. This is the level of spike present in the final digestate.

12.3.3.3   For a Post-Digestion Spike sample, the sample that was initially used for the Matrix Spike sample analysis shall be used for the Post-Digestion Spike analysis. Spike the unspiked aliquot of the undiluted digestate at two times the indigenous level or two times the CRQL, whichever is greater.

12.3.3.4   Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for Matrix Spike sample analysis. The EPA may require that a specific sample be used for the Matrix Spike sample analysis.

12.3.4   Calculations for Matrix Spike and Post-Digestion Spike Samples

12.3.4.1   If the Matrix Spike analysis is performed on the same sample that is chosen for the Duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.4). The average of the duplicate results cannot be used for the purpose of determining the %R.

12.3.4.2   Calculate the Matrix Spike and Post-Digestion Spike %R using the following equation:

EQ. 13 Matrix Spike and Post-Digestion Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,
SSR   =   Spiked Sample Result (µg/L or mg/kg) from EQ. 6 or EQ. 7

SR   =   Sample Result (original) (µg/L or mg/kg) from EQ. 6 or EQ. 7. When the sample concentration is less than the MDL, use SR=0.

SA   =   Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spiking amount used for the 'V_f' term and substituting the spiking standard concentration used for the 'C' term from EQ. 6 or EQ. 7.

NOTE: The units used for reporting SSRs will be identical to those used for reporting SRs.

12.3.5   Technical Acceptance Criteria for Matrix Spike and Post-Digestion Spike Samples

The Matrix Spike and Post-Digestion Spike %R shall be within the control limits of 75-125% (exception: Ag).

12.3.6   Corrective Action for Matrix Spike and Post-Digestion Spike Samples

12.3.6.1   If the Matrix Spike recovery is not at or within the limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with "*". An exception to this rule is granted when the sample concentration exceeds the SA concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the %R does not meet the 75-125% recovery criteria.

12.3.6.2   When the Matrix Spike recovery is outside the control limits and the sample result does not exceed four times the spike added, a Post-Digestion spike shall be performed for those analytes that do not meet the specified criteria (exception: Ag). Follow the procedures in Section 12.3.3.

12.3.6.3   If there is more than one Matrix Spike per matrix, per SDG, if one Matrix Spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

12.4 Duplicate Sample

12.4.1   Summary of Duplicate Sample

Duplicates are analyzed to help determine sample homogeneity and laboratory precision.

12.4.2   Frequency of Duplicate Sample

12.4.2.1   One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2] Duplicates cannot be averaged for reporting.

12.4.2.2   Duplicate sample analyses are not required for wipe samples.

12.4.3   Procedure for Duplicate Sample

12.4.3.1   Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. The EPA may require that a specific sample be used for duplicate sample analysis.

12.4.3.2   Prepare a second aliquot of the original sample. The duplicate sample shall be carried through the complete sample preparation procedure.

12.4.4   Calculations for Duplicate Sample

12.4.4.1   The Relative Percent Difference (RPD) for each analyte shall be calculated using the following equation:

EQ. 14 Duplicate Sample Relative Percent Difference

$$RPD = \frac{\left| S - D \right|}{(S+D)/2} \times 100$$

WHERE,
S   =   Sample Result (original) (μg/L or mg/kg) from EQ. 6 or EQ. 7
D   =   Duplicate Sample Result (μg/L or mg/kg) from EQ. 6 or EQ. 7

---

[2] The EPA may require additional duplicate sample analyses, upon request from the EPA Regional CLP COR.

12.4.5   Technical Acceptance Criteria for Duplicate Sample

12.4.5.1   The RPD shall be within the control limit of ±20 if the original and duplicate sample values are greater than or equal to five times the CRQL (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits).

12.4.5.2   The control limit shall be equal to the CRQL if either the sample or duplicate value are less than five times the CRQL or if one result is above five times the CRQL level and the other is below.

12.4.5.3   If both sample and duplicate values are less than the CRQL, the RPD is not recalculated.

12.4.6   Corrective Action for Duplicate Sample

12.4.6.1   If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*".

12.4.6.2   If there is more than one duplicate sample per matrix, per SDG, and if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG.

12.5 Laboratory Control Sample

12.5.1   Summary of Laboratory Control Sample

Aqueous/water, soil/sediment, and wipe Laboratory Control Samples (LCSs) shall be analyzed for each analyte using the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures employed for the samples received.

12.5.2   Frequency of Laboratory Control Sample

12.5.2.1   One LCS shall be prepared for each prepared batch of aqueous/water, soil/sediment, or wipe samples in an SDG.

12.5.2.2   If sufficient clean wipes are provided by the sampler, an additional LCS for the wipe samples shall be prepared by spiking a clean wipe.

12.5.3   Procedure for Laboratory Control Sample

The LCS for aqueous/water, soil/sediment, and wipe samples shall be prepared by spiking an aliquot of reagent water (50-100 mL for aqueous/water, 1 mL for soil/sediment and wipes) such that the final digestate shall contain each analyte at two times the CRQL for the associated matrix.

12.5.4   Calculations for Laboratory Control Sample

Calculate the results for LCS by using Equations 6, 7, or 8 as appropriate.

EQ. 15 LCS Percent Recovery

$$\%R = \frac{\text{Found (LCS)}}{\text{True (LCS)}} \times 100$$

```
WHERE,
Found (LCS)  = The found concentration of each analyte in
               the LCS (µg/L, mg/kg/ or µg) from EQs. 6, 7,
               or 8.  If the analyte concentration is less
               than the MDL, a value of zero shall be
               substituted for the Found (LCS).
True (LCS)   = Two times the CRQL for the appropriate
               matrix (µg/L, mg/kg, µg)
```

12.5.5   Technical Acceptance Criteria for Laboratory Control Sample

The %R shall be within the control limits of 70-130% for all analytes except Ag and Sb, for which the control limits are 50-150%.

12.5.6   Corrective Action for Laboratory Control Sample

12.5.6.1   If the %R for the LCS for aqueous/water or soil/sediment samples are outside the control limits of 70-130% (exception: Ag and Sb, control limits 50-150%), the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed with appropriate new QC.

12.5.6.2   If the %R for the LCS for wipes are outside the control limits of 70-130% (exception: Ag and Sb, control limits 50-150%), the Contractor shall note this in the SDG Narrative, since wipe samples are fully consumed by initial analysis and cannot be reprepared and reanalyzed.

12.6   ICP-AES Serial Dilution

12.6.1   Summary of ICP-AES Serial Dilution

The Contractor shall perform Serial Dilution analyses to check for interference effects.

12.6.2   Frequency of ICP-AES Serial Dilution

12.6.2.1   The ICP-AES serial dilution analysis shall be performed on a sample from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent, prior to reporting analyte concentration data.

12.6.2.2   Serial Dilution analysis is not required for wipe samples.

12.6.3   Procedure for ICP-AES Serial Dilution

12.6.3.1   Prepare an aliquot of the original sample digestate and dilute it 1:4 (five-fold) with 2% nitric acid. This dilution shall be analyzed as the serial dilution.

12.6.3.2   If the original sample requires dilution for any analyte, a 1:4 (five-fold) dilution of that dilution shall be prepared and analyzed as a serial dilution.

12.6.3.3   Samples identified as field blanks and PE samples shall not be used for serial dilution analysis.

12.6.4   Calculations for ICP-AES Serial Dilution

The percent difference for each component are calculated using the following equation:

EQ. 16 Serial Dilution Percent Difference

$$\%\text{Difference} = \frac{\left| I - S \right|}{I} \times 100$$

```
WHERE,
I     =   Initial sample result
S     =   Serial dilution result.  If the analyte concentration
          is less than the MDL, a value of zero shall be
          substituted for "S".
```

12.6.5   Technical Acceptance Criteria for ICP-AES Serial Dilution

If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), then the serial dilution (a five-fold dilution) shall be within 10% of the original determination after correction for dilution.

12.6.6   Corrective Action for ICP-AES Serial Dilution

12.6.6.1   If the dilution analysis for one or more analytes is not within a control limit of 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received and associated with that serial dilution must be flagged with an "*".

12.6.6.2   In the instance where there is more than one serial dilution per SDG, per matrix, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix in the SDG.

12.7   Method Detection Limit Determination

12.7.1   Before any field samples are analyzed, the MDLs shall be determined for each digestion procedure and instrument used prior to the start of contract analyses and annually thereafter. An MDL study shall also be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.7.1.1   To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.7.1.2   The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.7.1.3   The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

12.7.1.4   The MDLs for Hardness and TCLP are not required to be determined or reported.

12.8   Interelement Corrections

12.8.1   Before any field samples are analyzed, the IECs factors shall be determined prior to the start of contract analyses and at least annually thereafter following the procedures provided by the instrument manufacturer. Correction factors for spectral interference due to Al, Ca, Fe, and Mg shall be determined for all ICP-AES instruments at all wavelengths used for each analyte reported by ICP-AES. IEC factors shall also be reported for any other elements (including those on the TAL) that have been determined to interfere with the requested target analyte(s).

NOTE: Depending on sample matrix and interferences, it may be necessary to analyze IEC factors at a frequency greater than annually and/or at multiple concentrations comparable to the sample interferent levels.

12.8.2   If the instrument was adjusted in any way that may affect the ICP-AES IEC factors, the factors shall be redetermined and the results submitted for review.

12.8.3   All data used for the determination of the IEC factors shall be available to the EPA during an on-site laboratory evaluation.

12.8.4   Results from the IEC factors determination shall be reported for all ICP-AES analytes in accordance with Exhibit B – Reporting and Deliverables Requirements.

12.9   Summary of Quality Control Operations

The QC operations performed for ICP-AES analysis are summarized in Table 3 – QC Operations for ICP-AES.

13.0   METHOD PERFORMANCE

Not applicable.

14.0   POLLUTION PREVENTION

See Section 13.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

15.0   WASTE MANAGEMENT

See Section 14.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

16.0   REFERENCES

16.1   U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 200.7, Revision 4.4 (1994).

16.2   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 3050B, Revision 2, Third Edition, Update III, December 1996.

16.3   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Test Method 6010D, Revision 4, July 2014.

16.4   U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0   TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. INTERFERENT AND ANALYTE CONCENTRATIONS USED FOR ICP-AES
INTERFERENCE CHECK SAMPLE (ICS)

Analytes	(µg/L)	Interferents	(µg/L)
Ag	200	Al	250000
As	100	Ca	250000
Ba	500	Fe	100000
Be	500	Mg	250000
Cd	1000		
Co	500		
Cr	500		
Cu	500		
Mn	500		
Ni	1000		
Pb	50		
Sb	600		
Se	50		
Tl	100		
V	500		
Zn	1000		

NOTE: ICSA solution contains the interferents at the indicated
      concentrations. The ICSA solution may be analyzed at twice the
      concentration indicated when interferences are present at higher
      concentrations in the sample. The ICSAB solution contains all of
      the analytes and interferents listed above at the indicated
      concentrations.

TABLE 2. SPIKING LEVELS FOR MATRIX SPIKE SAMPLE ANALYSES

Analyte	Water (µg/L)	Soil[1] (mg/kg)
Al	2000	*
Sb	100	20
As	40	8
Ba	2000	400
Be	50	10
Cd	50	10
Ca	*	*
Cr	200	40
Co	500	100
Cu	250	50
Fe	1000	*
Pb	20	4
Mg	*	*
Mn	500	100
Ni	500	100
K	*	*
Se	100	20
Ag	50	10
Na	*	*
Tl	50	10
V	500	100
Zn	500	100

* No spike required.

[1] Concentrations in the spike sample when the dry weight of 1 gram of sample is taken for analysis. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values.

EQ. 17 Spiking Level Adjustment

$$mg/kg = µg/L \times \frac{\text{final volume (L)}}{\text{sample weight (g)}}$$

TABLE 3. QC OPERATIONS FOR ICP-AES

QC Operation	Frequency
Instrument Calibration	Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration for each wavelength used.
Continuing Calibration Verification	For each wavelength used, at a frequency of every 2 hours and at the beginning and end of each analytical sequence.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Blank	Every 2 hours and at the beginning and end of each analytical sequence. Performed immediately after the CCV.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Interference Check Sample	At the beginning of each analytical sequence after the ICB but before the CCV.
Matrix Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Digestion Spike	Each time Matrix Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Method Detection Limit Determination	Prior to start of contract analyses, annually thereafter, and after major instrument adjustment.
Interelement Corrections	Prior to start of contract analyses, annually thereafter, and after major instrument adjustment.

EXHIBIT D

INDUCTIVELY COUPLED PLASMA –
MASS SPECTROMETRY

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Exhibit D – Inductively Coupled Plasma – Mass Spectrometry

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Exhibit D – Inductively Coupled Plasma – Mass Spectrometry

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1.0   SCOPE AND APPLICATION

        This exhibit provides procedures for the use of Inductively Coupled
        Plasma - Mass Spectrometry (ICP-MS) to determine the concentration of
        total recoverable and dissolved elements in aqueous/water and
        soil/sediment samples taken from hazardous waste sites. All metals
        contained in the Inorganic Target Analyte List (TAL) for ICP-MS in
        Exhibit C - Inorganic Target Analyte List and Contract Required
        Quantitation Limits are quantitated by this method.

2.0   SUMMARY OF METHOD

2.1   General Method Overview

        This method describes the multi-element determination of trace elements
        by ICP-MS. Sample material in solution is introduced by nebulization
        into a radio-frequency plasma where energy transfer processes cause
        desolvation, atomization, and ionization. The ions are extracted from
        the plasma through a differentially pumped vacuum interface and
        separated on the basis of their mass-to-charge ratio. The separated
        ions are detected and the ion information processed by a data handling
        system. Interferences related to the technique must be recognized and
        corrected. Such corrections may include compensation for isobaric
        elemental interferences and interferences from polyatomic ions derived
        from plasma gas, reagents, or sample matrix. Instrumental drift, as
        well as suppressions or enhancements of instrument response, must be
        corrected for with the use of internal standards.

2.2   Summary of Digestion Procedures

2.2.1   Hotplate Acid Digestion of Aqueous/Water Samples (based on EPA
            Method 200.8)

2.2.2   Hotplate Acid Digestion of Soil/Sediment Samples (based on EPA
            Method 200.8)

3.0   DEFINITIONS

        See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0   INTERFERENCES

        Several types of interferences may contribute to inaccuracies in the
        determination of trace elements in aqueous/water and soil/sediment
        samples by ICP-MS. To prevent this, appropriate steps must be taken in
        all analyses to ensure that potential interferences are taken into
        account. Several types of interferences are given in Sections 4.1
        through 4.5 below.

4.1   Isobaric Elemental Interferences

        Isobaric elemental interferences are caused by isotopes of different
        elements which form singly or doubly charged ions of the same nominal
        mass-to-charge ratio, and which cannot be resolved by the mass
        spectrometer. All elements determined by this method have, at minimum,
        one isotope free of isobaric elemental interference. Of the analytical
        isotopes recommended for use with this method, only selenium-82 has an
        isobaric elemental interference (krypton-82). If alternative
        analytical isotopes having higher natural abundances are selected, in
        order to achieve greater sensitivity, an isobaric interference may
        occur. All data obtained under such conditions must be corrected by

measuring the signal from another isotope of the interfering element
and subtracting the appropriate signal ratio from the isotope of
interest. A record of this correction process shall be included with
the data. It should be noted that such corrections will only be as
accurate as the accuracy of the isotope ratio used in the elemental
equation for data calculations. Relevant isotope ratios should be
established prior to the application of any corrections. Interferences
from doubly charged ions may not be correctable. The Contractor shall
monitor the intensities of the singly charged ions of those isotopes
that can cause doubly charged interferences and note high readings in
the Sample Delivery Group (SDG) Narrative.

4.2   Abundance Sensitivity

Abundance sensitivity is a property defining the degree to which the
wings of a mass peak contribute to adjacent masses. Abundance
sensitivity is affected by ion energy and mass filter operating
pressure. Wing overlap interferences may result when a small ion peak
is being measured adjacent to a large one. The potential for these
interferences should be recognized and the spectrometer resolution
should be adjusted to minimize them.

4.3   Isobaric Polyatomic Ion Interferences

Isobaric polyatomic ion interferences are caused by ions consisting of
more than one atom which have the same nominal mass-to-charge ratio as
the isotope of interest, and which cannot be resolved by the mass
spectrometer. These ions are commonly formed in the plasma or
interface system from support gases or sample components. Most of the
common interferences have been identified and are listed in Table 2 -
Isobaric Molecular-Ion Interferences, with the target analytes
affected. Such interferences must be recognized, and when they cannot
be avoided by the selection of alternative analytical isotopes,
appropriate corrections must be made to the data. The use of collision
cells to reduce these interferences is permitted. Equations for the
correction of data should be established at the time of the analytical
sequence, since polyatomic ion interferences will be highly dependent
on the sample matrix and chosen instrument conditions.

4.4   Physical Interferences

Physical interferences are associated with the physical processes which
govern the transport of the sample into the plasma, sample conversion
processes in the plasma, and the transmission of ions through the
plasma-mass spectrometer interface. These interferences may result in
differences between instrument responses for the sample and the
calibration standards. Physical interferences may occur in the
transfer of solution to the nebulizer (e.g., viscosity effects), at the
point of aerosol formation and transport to the plasma (e.g., surface
tension), or during the excitation and ionization processes within the
plasma itself. High levels of dissolved solids in the sample may
contribute to deposits of material on the extraction and/or skimmer
cones. Deposits can reduce the effective diameter of the orifices and
therefore ion transmission. Dissolved solid levels not exceeding 0.2%
(w/v) have been recommended to reduce such effects. Internal
standardization may be effectively used to compensate for many physical
interference effects. Internal standards ideally should have similar
analytical behavior to the elements being determined.

4.5   Memory Interferences

Memory interferences result when isotopes of elements in a previous
sample contribute to the signals measured in a new sample. Memory
effects, or carryover, can result from sample deposition on the sampler
and skimmer cones, as well as from the buildup of sample material in
the plasma torch and spray chamber. The site where these effects occur
is dependent on the element and can be minimized by flushing the system
with a rinse solution between samples (Section 7.3.3). The possibility
of memory interferences should be recognized within an analytical
sequence and suitable rinse times or monitoring should be used to
reduce them. Memory interferences may also be assessed within an
analytical sequence by using a minimum of three replicate integrations
for data acquisition. If the integrated signal values drop
consecutively, the analyst should be alerted to the possibility of a
memory effect, and should examine the analyte concentration in the
previous sample to identify if it was high. If a memory interference
is suspected, the sample should be reanalyzed after a rinse period.

5.0   SAFETY

See Section 12.0 in Exhibit D - Introduction to Inorganic Analytical
Methods.

6.0   EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes
only. No endorsement is implied. Equivalent performance may be
achieved using equipment and supplies other than those specified here;
however, a demonstration of equivalent performance meeting the
requirements of this Statement of Work (SOW) is the responsibility of
the Contractor. The Contractor shall document any use of alternate
equipment or supplies in the SDG Narrative.

6.1   Glassware/Labware

6.1.1   250 milliliter (mL) beaker or other appropriate vessel (glass or
         plastic)

6.1.2   Watch glasses (glass or plastic)

6.1.3   Funnels

6.1.4   Graduated cylinders

6.1.5   Various volumetric flasks (Class A) and calibrated pipettes.
         Manufacturer's instructions should be followed for the calibration
         and maintenance of adjustable pipettes.

6.1.6   Thermometer that covers range of 0-200°C

6.1.7   Whatman No. 42 filter paper (or equivalent)

6.1.8   Hotplate, block digester, or other heating source capable of
         maintaining 95°C (±3°C)

6.1.9   Balances – Top loader balance, 300 gram (g) capacity, and a minimum
         sensitivity of ±1.0 milligram (mg)

         A balance calibration must be checked with known masses once per
         each day of use. This verification consists of a check with two
         weights covering the range expected (approximately ±50% of the
         expected measured mass) for each balance and be accurate to ±1.0 mg.
         The masses that are used to check the balances daily must be checked
         on a monthly basis using National Institute of Standards and

Technology (NIST)-traceable known reference masses (Class '1' or '2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified every five years or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates that these criteria have been met.

6.2   Inductively Coupled Plasma - Mass Spectrometer

The ICP-MS consists of:

- An instrument capable of scanning the mass range 5-250 atomic mass units (u) with a minimum resolution capability of 1 u peak width at 5% peak height and either a conventional or extended dynamic range detector.

- A radio-frequency generator compliant with Federal Communications Commission (FCC) regulations.

- A high purity (99.99%) argon gas supply.

- A variable speed peristaltic pump to deliver sample solution to the nebulizer.

- A mass-flow controller on the nebulizer gas supply is required.

7.0   REAGENTS AND STANDARDS

7.1   Reagents

Reagents may contain elemental impurities that might affect the integrity of analytical data. Due to the high sensitivity of ICP-MS, high-purity reagents should be used whenever possible. Suitable acids are available from a number of manufacturers or may be prepared by sub-boiling distillation. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid is used; however, it should be noted that hydrochloric acid is required to maintain stability in solutions containing antimony and silver. When hydrochloric acid is used, corrections for the chloride polyatomic ion interferences must be applied to all data.

7.1.1   Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions.

7.1.2   Hydrochloric acid - Concentrated 32-38% (specific gravity 1.19).

7.1.3   Hydrochloric acid (50% v/v) - Add 500 mL conc. hydrochloric acid to 400 mL reagent water and dilute to 1 Liter (L).

7.1.4   Nitric acid - Concentrated 67-70% (specific gravity 1.41).

7.1.5   Nitric acid (50% v/v) - Add 500 mL conc. nitric acid to 400 mL reagent water and dilute to 1 L.

7.1.6   Nitric acid (2% v/v) - Add 20 mL conc. nitric acid to 400 mL reagent water and dilute to 1 L.

7.1.7   Nitric acid (1% v/v) - Add 10 mL conc. nitric acid to 400 mL reagent water and dilute to 1 L.

7.2   Standards

7.2.1   Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Inorganic Analytical Methods, Section 11.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Samples, sample digestates, and standards must be stored separately.

7.2.2   Stock Standard Solutions

Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals (at least 99.99% pure). All salts must be dried for 1 hour at 105°C unless otherwise specified. Stock solutions should be stored in Fluorinated Ethylene Propylene (FEP) fluorocarbon bottles. Note that some metals, particularly those which form surface oxides, require cleaning prior to being weighed. This may be achieved by pickling the surface of the metal in acid. An amount in excess of the desired weight should be pickled repeatedly, rinsed with reagent water, dried, and weighed until the desired weight is achieved.

7.2.3   Secondary Dilution Standards

Prepare mixed secondary dilution standard solutions by diluting the appropriate volumes of stock standards with 1% (v/v) nitric acid, or as recommended by the manufacturer, to obtain the final volume. Originating stock standards should be checked for the presence of impurities which might influence the accuracy of the standard. Freshly prepared standards should be transferred to acid-cleaned, not previously used, FEP fluorocarbon bottles for storage and monitored periodically for stability. Mixed secondary dilution standard solutions may be purchased. The purchased standards shall meet the requirements in Section 7.2.1.

7.2.4   Working Standards

7.2.4.1   Mixed Calibration Standard Solutions

Care must be taken in the preparation of mixed calibration standards to ensure that the analytes are compatible and stable. Fresh calibration standards should be prepared from mixed standard solutions every 2 weeks or less.

Prepare the mixed standards to levels appropriate to the operating range of the instrument using 1% (v/v) nitric acid or to match the matrix of the digested samples. The analyte concentrations in the calibration standards should be sufficient to produce good measurement precision and to accurately define the slope of the response curve.

7.2.4.2   Internal Standard Solution

The internal standard solution is to be added to all digested samples, blanks, and standards by the analyst prior to analysis, or it can be added automatically by the instrument during analysis of all digested samples, blanks, and standards. Prepare the mixed internal standard solution by following the manufacturer's guidelines.

7.2.4.3   Tuning Solution

This solution is used for instrument tuning and mass calibration prior to analysis. Prepare a mixed standard by diluting beryllium, magnesium, cobalt, indium, and lead stock standards to 100 micrograms/Liter (µg/L) with 1% (v/v) nitric acid. The concentration of this solution can be reduced based on recommendations from the instrument manufacturer. If indium is also selected as an internal standard, and added automatically, the resulting indium concentration in the tune solution reaching the instrument may exceed 100 µg/L and is allowed for indium only.

7.2.4.4   Interference Check Sample Solution

7.2.4.4.1   The Interference Check Sample (ICS) consists of two solutions: ICS Solution A (ICSA) and ICS Solution AB (ICSAB). ICSA consists of the interferents and ICSAB consists of the analytes mixed with the interferents. The ICS standards (ICSA for the interferents only and ICSB for the analytes only) shall be obtained by the EPA.

7.2.4.4.1.1   Only if the ICS solutions are not available from the EPA, ICSs shall be prepared with interferent and analyte concentrations at the levels specified in Table 1 – Interferent and Analyte Concentrations Used for ICP-MS Interference Check Sample (ICS).

7.2.4.4.1.2   If the solutions are prepared, the mean value and standard deviation shall be established by initially analyzing the ICSs at least five times repetitively for each analyte listed. Results shall be within the control limits of ±20% of the established mean value or ±2 times the analyte's Contract Required Quantitation Limits (CRQL) of the established mean value, whichever is greater. The mean and standard deviation shall be reported in the raw data.

7.2.4.5   Initial Calibration Verification Solutions

7.2.4.5.1   The Initial Calibration Verification (ICV) solution(s) shall be obtained from the EPA.

7.2.4.5.1.1   If the solution(s) is (are) not available from the EPA, then the ICV solution shall be prepared by the laboratory using a certified solution for each analyte from an independent source. An independent source is defined as a standard composed of the analytes from a different source than those used in the standards for the instrument calibration. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range.

7.2.4.5.1.2   The ICV standard shall be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier.

7.3   Blanks

Three types of blanks are required for this method. A Calibration Blank is used to establish the analytical calibration curve, a Preparation Blank (see Section 12.1) is used to assess possible contamination from the sample preparation procedure and to assess spectral background, and a rinse solution is used to flush the instrument between samples to reduce memory interferences.

7.3.1   Calibration Blank - Consists of 1% (v/v) nitric acid in reagent water or matrix matched to the digested samples. The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are identical in composition to the Calibration Blank and are used to monitor for analyte carryover.

7.3.2   Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Preparation Blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Soil/sediment blanks shall use 1.00 mL (±0.01 mL) of reagent water.

7.3.3   Rinse Solution – Must contain sufficient nitric acid to allow the instrument to return to baseline between the analysis of digested samples, blanks, and standards. The rinse solution would typically consist of 2% (v/v) nitric acid in reagent water.

8.0   SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1   Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All soil/sediment samples must be iced or refrigerated at ≤6°C but not frozen from the time of collection until receipt at the laboratory.

8.2   Sample Storage

All aqueous/water and soil/sediment samples must be stored at ≤6°C but not frozen from the time of sample receipt until digestion. If aqueous/water samples are received in glass containers, the Contractor shall note this in the SDG Narrative.

8.2.1   Unused Sample Storage

The Contractor shall retain the unused portion of aqueous/water and soil/sediment samples, for a period of 60 days after delivery of a complete, reconciled data package to the EPA. The samples may be stored at room temperature.

8.2.2   Digestate Sample Storage

Sample digestates must be stored until 180 days after delivery of a complete, reconciled data package to the EPA. The Contractor shall store sample ICP-MS digestates in plastic bottles. The bottles shall be labeled with the EPA Sample Number, Case Number, SDG Numbers, MA No. (if applicable), and digestion date. A logbook of stored digestates, listing the EPA Sample Numbers and associate Case and SDG Numbers, shall be maintained.

8.2.3   Container Storage

The Contractor shall retain the empty sample containers for 60 days after the delivery of a complete, reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.3   Contract Required Holding Time

The holding time for metals is 180 days from Validated Time of Sample Receipt (VTSR).

9.0   CALIBRATION AND STANDARDIZATION

9.1   Instrument Operating Parameters

Because of the differences between various manufacturers and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL), precision, and interference effects must be investigated and established for each individual analyte on that particular instrument. All measurements must be within the operational range of the instrument where corrections are valid. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used to satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2   Pre-Calibration Routine

Prior to calibration, the Contractor shall set up each instrument with the operating parameters established in Section 9.1 and conduct any necessary adjustments to the plasma conditions. The instrument must be allowed to become stable (usually 30 minutes) before calibration is performed.

9.3   Instrument Performance Check

9.3.1   Summary of Instrument Performance Check

The Contractor shall demonstrate instrument stability and precision by analyzing the tuning solution or Instrument Performance Check (IPC) sample.

9.3.2   Frequency of Instrument Performance Check

The tuning solution shall be analyzed prior to instrument calibration.

9.3.3   Procedure for Instrument Performance Check

The Contractor shall analyze the tuning solution as a single analysis with at least five integrations.

9.3.4   Calculations for Instrument Performance Check

The Percent Relative Standard Deviation (%RSD) shall be calculated by the instrument manufacturer's software.

9.3.5   Technical Acceptance Criteria for Instrument Performance Check

9.3.5.1   The mass calibration shall be within 0.1 u over the range of 6 to 210 u. The peak width shall be measured at the height set by the instrument manufacturer.

9.3.5.2   The %RSD shall be less than or equal to 5.0% for each isotope in the tuning solution.

9.3.5.3   The Contractor shall report the full peak width and the percentage of peak height this full peak width (in u) was measured at for each of the isotope masses in the tuning solution.

9.3.6   Corrective Action for Instrument Performance Check

9.3.6.1   If the mass calibration is not within 0.1 u over the range of 6 to 210 u, the analysis shall be terminated, the problem corrected, and the instrument re-tuned.

9.3.6.2   If the %RSD exceeds 5.0%, the analysis shall be terminated, the problem corrected, and the instrument re-tuned.

9.3.6.3   No sample results may be reported from an analytical sequence associated with a tune that does not meet the technical acceptance criteria.

9.4   Instrument Calibration Procedure

9.4.1   Summary of Instrument Calibration

Prior to sample analysis, the Contractor shall calibrate each instrument to determine sensitivity and linearity of the response.

9.4.2   Frequency of Instrument Calibration

Each instrument shall be calibrated each time it is turned on, set up, or after ICV, ICB, Continuing Calibration Verification (CCV), or CCB failure. The instrument calibration date and time shall be included in the raw data.

9.4.3   Procedure for Instrument Calibration

9.4.3.1   Each instrument shall be calibrated according to the manufacturer's recommended procedures.

9.4.3.2   At least six calibration standards shall be used for each analyte. The calibration standards shall be prepared as in Section 7.2.4.1. One of the standards shall be a blank standard and one shall be at or below the CRQL but greater than the MDL. The rest of the standards shall be uniformly spread out in graduated amounts over the appropriate calibration range of the analyte.

9.4.3.3   A minimum of three replicate integrations are required for data acquisition. The Contractor shall use the average of all the integrations for instrument calibration and data reporting.

9.4.4   Calculations for Instrument Calibration

9.4.4.1   The calibration curve shall be calculated for each analyte using linear regression by plotting the concentration of the standard (in µg/L) on the X-axis versus the corrected instrument response on the Y-axis. The corrected instrument responses are those corrections (e.g., correction for background, internal standards, interferences, calibration blank) that may be applied to the raw uncorrected instrument response prior to determining the calibration curve.

9.4.4.2   The Contractor may use standard linear regression, weighted linear regression (1/concentration, 1/square of concentration, variance, 1/variance, standard deviation, 1/standard deviation, or 1/square of standard deviation), or linear regression with zero force calibration models, as appropriate, for the above calculation. No other types of equations (e.g., quadratic) are to be used.

9.4.4.3   The calibration curve for each analyte shall be checked to establish the representativeness of the data that were used to produce it. This check involves re-fitting the data of the non-blank calibration standards back to the calibration curve, and

computing the difference between the calculated concentration and the expected concentration for each of these standards. The difference is then divided by the expected concentration of the respective standard and multiplied by 100.

9.4.5   Technical Acceptance Criteria for Instrument Calibration

9.4.5.1   The correlation coefficient of the calibration curve shall be greater than or equal to 0.995.

9.4.5.2   The Percent Difference (%D) for each of the standards shall be within the control limits of ±30%.

9.4.5.3   If a standard is analyzed for a particular analyte at a concentration that is below the CRQL and the %D criteria is not met, that standard can be excluded from the calibration curve as long as the lowest non-zero standard for that analyte is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.4.6   Corrective Action for Instrument Calibration

9.4.6.1   Sample analysis shall not begin until the criteria described in Section 9.4.5 have been met.

9.4.6.2   Any changes or corrections to the analytical system shall be followed by recalibration.

9.5   Initial Calibration Verification

9.5.1   Summary of Initial Calibration Verification

Prior to sample analysis, the Contractor shall demonstrate the accuracy of the instrument calibration for each instrument through the analysis of an ICV standard.

9.5.2   Frequency of Initial Calibration Verification

The ICV shall be analyzed immediately after the instrument has been calibrated.

9.5.3   Procedure for Initial Calibration Verification

9.5.3.1   The ICV shall be analyzed at each mass used to report final results for each analyte.

9.5.3.2   The ICV shall reflect the conditions of analysis of the associated analytical samples.

9.5.4   Calculations for Initial Calibration Verification

9.5.4.1   The Percent Recovery (%R) of the ICV shall be calculated using the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{\text{Found (ICV)}}{\text{True (ICV)}} \times 100$$

WHERE,
Found (ICV)   = The found concentration of the analyte in the ICV Solution
True (ICV)   = The expected concentration of the analyte in the ICV Solution

9.5.4.2   The Percent Relative Standard Deviation (%RSD) from all replicate integrations shall be calculated for each mass used to report final results using Equations 2, 3, and 4.

EQ. 2 Percent Relative Standard Deviation Calculation

$$\%RSD = \frac{SD}{\overline{X}} \times 100$$

WHERE,

SD   = Standard deviation of ICV replicates (per analyte) from EQ. 3

$\overline{X}$ = Mean value of the ICV replicates (per analyte) from EQ. 4

9.5.4.3   Equation 3 is the general formula for Standard Deviation (SD) for a statistically small set of values.

EQ. 3 Standard Deviation Calculation

$$SD = \sqrt{\frac{\sum\limits_{i=1}^{n} (X_i - \overline{X})^2}{(n-1)}}$$

WHERE,
X_i = Each individual value used to calculate the mean
$\overline{X}$ = The mean of n values from EQ. 4
n   = Total number of values

9.5.4.4   Equation 4 is the general formula for the mean of a set of values ($\overline{X}$).

EQ. 4 Mean Value Calculation

$$\overline{X} = \frac{\sum\limits_{i=1}^{n} X_i}{n}$$

WHERE,
X_i = Each individual value used to calculate the mean
n   = Total number of values

9.5.5   Technical Acceptance Criteria for Initial Calibration Verification

9.5.5.1   The ICV %R shall be within the control limits of 90-110%.

9.5.5.2   The %RSD of the ICV integrations shall be less than or equal to 5.0%.

9.5.6   Corrective Action for Initial Calibration Verification

If the recovery is outside the control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

9.6   Continuing Calibration Verification

9.6.1   Summary of Continuing Calibration Verification

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration through the analysis of a CCV standard. The Contractor shall not analyze a blank immediately prior to the analysis of the CCV.

9.6.2   Frequency of Continuing Calibration Verification

9.6.2.1   A CCV shall be analyzed at the beginning of the analytical sequence and after the last analytical sample. A CCV shall also be analyzed at a frequency not to exceed every 2 hours during an analytical sequence. See the example analytical sequence in Section 10.3.6.

9.6.2.2   The analytical sequence can continue indefinitely as long as samples are being continuously analyzed without the instrument being turned off and successive CCV standards meet the technical acceptance criteria in Section 9.6.5.

9.6.3   Procedure for Continuing Calibration Verification

9.6.3.1   The CCV standard shall be prepared using the same source and in the same acid matrix as the calibration standards by combining compatible analytes at a concentration at or near the mid-level of their respective calibration curve.

9.6.3.2   The same CCV standard shall be used throughout the analytical sequences for an SDG.

9.6.3.3   The CCV shall be analyzed at each mass used to report final results for each analyte.

9.6.3.4   Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.6.4   Calculations for Continuing Calibration Verification

9.6.4.1   The %R of the CCV shall be calculated using the following equation:

EQ. 5 CCV Percent Recovery

$$\%R = \frac{\text{Found (CCV)}}{\text{True (CCV)}} \times 100$$

WHERE,
Found (CCV)   = The found concentration of the analyte in the CCV Solution
True (CCV)   = The expected concentration of the analyte in the CCV Solution

9.6.4.2   The %RSD from all replicate integrations shall be calculated for each mass used to report final results using Equations 2, 3, and 4 above.

9.6.5   Technical Acceptance Criteria for Continuing Calibration Verification

9.6.5.1   The CCV %R shall be within the control limits of 90-110%.

9.6.5.2   The %RSD of the CCV integrations shall be less than or equal to 5.0%.

9.6.5.3   All samples shall be analyzed within 2 hours of an acceptable opening and closing CCV.

9.6.6   Corrective Action for Continuing Calibration Verification

If the deviations of the CCV are greater than the specified control limits of 90% Recovery (low) or 110% Recovery (high), or if the %RSD as calculated from all replicate integrations exceeds 5.0%, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed for the analytes affected.

9.7   Initial and Continuing Calibration Blank

9.7.1   Summary of Calibration Blank

Prior to sample analysis, periodically during analysis, and at the conclusion of the analysis, the Contractor shall demonstrate the stability of the calibration baseline through the analysis of an ICB and CCB.

9.7.2   Frequency of Calibration Blank

9.7.2.1   An ICB shall be analyzed immediately after the instrument has been calibrated and the calibration verified by the analysis of the ICV.

9.7.2.2   Each CCB shall be analyzed immediately after the analysis of a CCV.

9.7.3   Procedure for Calibration Blank

9.7.3.1   The ICB and CCB samples shall be analyzed at each mass used for reporting final results for each analyte.

9.7.3.2   The ICB analyzed shall reflect the conditions of analysis of the associated analytical samples.

9.7.3.3   Each CCB analyzed shall reflect the conditions of analysis of the associated analytical samples (the preceding analytical samples up to the previous CCB as applicable and the analytical samples following this CCB up to the next CCB as applicable).

9.7.4   Calculations for Calibration Blank

The results for the ICB and CCB samples shall be calculated using Equation 6 in Section 11.0.

9.7.5   Technical Acceptance Criteria for Calibration Blank

The absolute value of each calibration blank result shall be less than or equal to the CRQL for aqueous/water samples for the analyte.

9.7.6   Corrective Action for Calibration Blank

If the absolute value of the calibration blank exceeds the CRQL for aqueous/water samples, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all affected analytical samples analyzed since the last compliant calibration blank performed for the analytes affected.

10.0 PROCEDURE

10.1 Aqueous/Water Sample Preparation

Preparation Method 200.8 – Total Recoverable Analytes [based on the EPA NERL Method 200.8, Revision 5.5 (October 1999)]

10.1.1   If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.

10.1.2   For the determination of total recoverable analytes in aqueous/water samples, transfer a 100 mL (±1 mL) aliquot from a well-mixed, acid-preserved sample to an appropriately sized (approximately 250 mL) digestion vessel (e.g., a beaker or hot block digestion tube). The sample shall not be diluted prior to digestion.

NOTE: A reduced sample volume of 50 mL can be used. If this reduced volume is used, then all other reagents and volumes shall be reduced appropriately.

10.1.3   Add 2 mL 50% (v/v) nitric acid and 1 mL 50% (v/v) hydrochloric acid to the beaker containing the measured volume of sample. Place the beaker on a hotplate, or other comparable heating device, for solution evaporation. The hotplate should be located in a fume hood and previously adjusted to provide evaporation at a temperature of 95°C (±3 °C), when covered. The beaker should be covered with an elevated watch glass or other necessary steps should be taken to prevent sample contamination from the fume hood environment.

10.1.4   Reduce the volume of the sample aliquot to about 20 mL by gently heating at 95°C (±3°C). **DO NOT BOIL**. This step takes about 2 hours for a 100 mL aliquot with the rate of evaporation rapidly increasing as the sample volume approaches 20 mL. (A spare beaker containing 20 mL of water can be used as a gauge.)

10.1.5   Cover the beaker with a watch glass to reduce additional evaporation and gently reflux the sample for 30 minutes. (Slight boiling may occur, but vigorous boiling must be avoided to prevent loss of the $HCl-H_2O$ azeotrope.)

10.1.6   Allow the beaker to cool. Quantitatively transfer the sample solution to a 100 mL volumetric flask, make to volume with reagent water, stopper, and mix.

10.1.7   Allow any undissolved material to settle overnight, or centrifuge or filter a portion of the prepared sample until clear to avoid plugging the nebulizer with solid particles.

10.1.8   The sample is now ready for analysis. Because the effects of various matrices on the stability of the samples cannot be characterized, all analyses should be performed as soon as possible after the completed preparation.

10.1.8.1   The digested sample may be further diluted if high levels of interferences (e.g., chloride) are noted or if any of the analytes exceed the upper limit of their respective calibration curves, according to Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0.

10.2 Soil/Sediment Sample Preparation

Preparation Method 200.8 – Total Recoverable Analytes [based on the EPA NERL Method 200.8, Revision 5.5 (October 1999)]

10.2.1   Mix the sample thoroughly to achieve homogeneity. Weigh to the nearest 0.01 g and transfer 1.0 – 1.5 g sample (wet weight) to an appropriately sized digestion vessel (e.g., a beaker or hot block digestion tube).

10.2.2   Add 4 mL of 50% (v/v) nitric acid and 10 mL of 1:4 HCl, mix the slurry, and cover with a watch glass. Heat the sample to 95°C (±3°C) and reflux for 30 minutes without boiling.

10.2.3   After cooling, transfer the digestate to a 100 mL volumetric flask and dilute to volume with reagent water, stopper, and mix. Allow the sample extract solution to stand overnight to separate insoluble material or centrifuge a portion of the sample until clear. The solution being analyzed must be clear to avoid plugging the nebulizer with solid particles. In place of settling, a portion of the sample (after dilution and mixing) may be filtered through Whatman No. 42 filter paper (or equivalent). Care should be taken to avoid potential contamination from filtration.

10.2.4   Prior to analysis, adjust the chloride concentration by pipetting 20 mL of the prepared solution into a 100 mL volumetric flask, dilute to volume with reagent water, and mix.

10.2.5   The sample is now ready for analysis. Report the final volume as 500 mL and the dilution factor as 1.0 for samples not requiring any additional dilution beyond that specified for chloride adjustment.

10.2.5.1   The digested sample may be further diluted if high levels of interferences are noted, high dissolved solid content, or if any of the analytes exceed the upper limit of their respective calibration curves, according to Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0.

10.3 Sample Analysis

10.3.1   It is highly recommended that a semi-quantitative analysis be carried out to screen for high element concentrations. This screening procedure can be performed using Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) or some other technique. Information gained from this may be used to prevent potential damage to the detector during sample analysis and to identify elements which may be higher than the calibrated range. Matrix screening may be carried out by diluting the sample by a factor of 500 and analyzing in semi-quantitative mode. The sample should also be screened for background levels of all elements chosen for use as internal standards in order to prevent bias in the calculation of analytical data. Undiluted sample results are not required if elements are present in the undiluted sample digestate at levels which could damage the detector.

10.3.2   For all sample analyses, a minimum of three replicate integrations are required for data acquisition. Use the average of all the integrations for data reporting.

10.3.3   In accordance with the instrument manufacturer's instructions, a rinse solution should be used to flush the system between samples. Allow sufficient time to remove traces of the previous sample. Samples should be aspirated for a sufficient period of time to obtain a stable response prior to the collection of data.

10.3.4   Sample digestates having high levels of interferences or concentrations higher than the established calibrated range as determined by the expected concentration of the highest calibration standard shall be diluted into range and reanalyzed, according to Exhibit D – Introduction to Inorganic Analytical Methods, Section 7.0. The sample digestate should first be analyzed for the trace elements, protecting the detector from the high concentration elements, if necessary, by the selection of appropriate scanning windows. The sample digestate should then be diluted for the determination of the remaining elements.

10.3.5   All masses which might affect data quality must be monitored during the analytical sequence. At a minimum, those masses identified in Table 3 - Recommended Isotopes and Masses for Selected Elements, must be monitored in the same scan that is used for the collection of the data. This information should be used to correct the data for identified interferences.

10.3.6   Example Analytical Sequence for ICP-MS Including the Instrument Calibration:

Tune
S##
S##
S##
S##
S##
S##
ICV
ICB
ICSA
ICSAB
CCV###
CCB###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Recommended Elemental Equations

Elemental expressions recommended for sample data calculations are listed in Table 4 – Recommended Elemental Expressions for Isobaric Interferences. Do not report element concentrations below the MDL.

11.2 Data Value Corrections

Data values as produced by the instrument should be corrected for instrument drift or sample matrix induced interferences by the application of internal standardization. Corrections for characterized spectral interferences should be applied to the data. Chloride interference corrections should be made on all samples, regardless of the addition of hydrochloric acid, as the chloride ion is a common constituent of environmental samples.

11.3 Multiple Monitored Isotopes

If an element has more than one monitored isotope, examination of the concentration calculated for each isotope or the isotope ratios will provide useful information in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of sample concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes; therefore, differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes.

11.4 Calculate Target Analyte Concentrations

Calculate the Target Analyte concentration using the following equations.

11.4.1 Aqueous/Water Sample Calculation

EQ. 6 Aqueous/Water Sample Concentration

$$\text{Concentration } (\mu g/L) = C \times \frac{V_f}{V} \times DF$$

WHERE,
C   =   Instrument value in µg/L (The average of all replicate integrations)
V_f =   Final digestion volume (mL)
V   =   Initial aliquot amount (mL)
DF =   Dilution Factor

11.4.2 Soil/Sediment Sample Calculation

The concentrations determined in the digestate are to be reported on the basis of the dry weight of the sample, in units of mg/kg:

EQ. 7 Soil/Sediment Sample Concentration

$$\text{Concentration (mg/kg dry weight)} = C \times \frac{V_f}{W \times S} \times DF \, / \, 1000$$

```
WHERE,
C    =    Instrument value in µg/L (The average of all replicate
          integrations)
Vf   =    Final digestion volume (mL)
W    =    Initial aliquot amount (g)
S    =    % Solids/100 (Exhibit D – General Inorganic Analysis,
          Section 10.1.1)
DF   =    Dilution Factor
```

11.5 Adjusted Contract Required Quantitation Limit Calculation

11.5.1   Calculate the adjusted CRQL for aqueous/water samples, by multiplying the CRQL (µg/L) by the sample dilution factor and the V_f/V term as noted in Equation 6.

11.5.2   Calculate the adjusted CRQL for soil/sediment samples using the following equation:

EQ. 8 Adjusted Soil/Sediment CRQL

$$\text{Adjusted CRQL(mg/kg)} = C \times \frac{W_M}{W \times S} \times \frac{V_f}{V_M} \times DF$$

```
WHERE,
C    =    CRQL (mg/kg)
WM   =    Minimum method required aliquot amount (g) (1.00 g)
W    =    Initial aliquot amount (g)
VM   =    Method required final sample digestion volume (mL) (100 mL)
Vf   =    Final digestion volume (mL)
S    =    % Solids/100 (Exhibit D – General Inorganic Analysis,
          Section 10.1.1)
DF   =    Dilution Factor
```

12.0 QUALITY CONTROL

12.1 Preparation Blank Sample

12.1.1   Summary of Preparation Blank Sample

The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.1.2   Frequency of Preparation Blank Sample

At least one Preparation Blank shall be prepared with every SDG, or with each preparation batch, whichever is more frequent.

12.1.3   Procedure for Preparation Blank Sample

The Preparation Blank shall be carried through the complete sample preparation procedure for the matrix and contain the same acid concentration in the final digestate as the samples.

12.1.4   Calculations for Preparation Blank Sample

Calculate the results for aqueous Preparation Blanks by using Equation 6. Calculate the results for soil/sediment Preparation Blanks by using Equation 7.

12.1.5   Technical Acceptance Criteria for Preparation Blank Sample

12.1.5.1   The absolute value of the Preparation Blank result shall be less than or equal to the CRQL.

12.1.5.2   Any analyte concentration in the Preparation Blank may be greater than the CRQL, if the concentration of the analyte in the associated samples is greater than or equal to 10 times the blank concentration.

12.1.5.3   Any analyte concentration in the Preparation Blank may be less than the negative CRQL, if the concentration in the associated samples is greater than or equal to 10 times the CRQL.

12.1.6   Corrective Action for Preparation Blank Sample

12.1.6.1   If any analyte concentration in the Preparation Blank is greater than the CRQL, and the concentration of the analyte in any of the associated samples is less than 10 times the blank concentration, then all samples with less than 10 times the blank concentration shall be reprepared and reanalyzed with appropriate new QC for that analyte.

12.1.6.2   If any analyte concentration in the Preparation Blank is less than the negative CRQL, and the concentration in the associated samples is less than 10 times the CRQL, then all samples with less than 10 times the CRQL concentration shall be reprepared and reanalyzed with appropriate new QC for that analyte.

12.2   Interference Check Sample

12.2.1   Summary of Interference Check Sample

The Contractor shall analyze the Interference Check Sample to verify elemental and polyatomic corrections. If not available, then the ICS can be prepared by the analyst.

12.2.2   Frequency of Interference Check Sample

The Contractor shall analyze, quantitate, and report the results for all elements on the TAL, and monitor for all interferents, including those caused by these elements, immediately after the initial calibration sequence, but not before the ICV/ICB.

12.2.3   Procedure for Interference Check Sample

12.2.3.1   The ICS solutions (Section 7.2.4.4) shall be analyzed according to the instructions supplied with the ICS.

12.2.3.2   The Contractor shall initially analyze the ICSA and the ICSAB undiluted. Any dilution of the ICS (for the highest concentration elements) necessary to meet the calibrated range values of the instrument shall be analyzed after the undiluted analyses of the ICSA and ICSAB.

12.2.3.3   An ICS analysis consists of analyzing both solutions consecutively, starting with Solution A.

12.2.4   Calculations for Interference Check Sample

12.2.4.1   The ICSA/ICSAB sample concentrations shall be calculated using Equation 6.

12.2.4.2   The %R of the ICSA and ICSAB shall be calculated using the following equations:

EQ. 9 ICSA Percent Recovery

$$\%R = \frac{\text{Found (ICSA)}}{\text{True (ICSA)}} \times 100$$

WHERE,
Found (ICSA) = The found concentration of the analyte in the
                ICSA Solution
True (ICSA)   = The expected concentration of the analyte in the
                ICSA Solution

EQ. 10 ICSAB Percent Recovery

$$\%R = \frac{\text{Found (ICSAB)}}{\text{True (ICSAB)}} \times 100$$

WHERE,
Found (ICSAB) = The found concentration of the analyte in the
                  ICSAB Solution
True (ICSAB)   = The expected concentration of the analyte in
                  the ICSAB Solution

12.2.5   Technical Acceptance Criteria for Interference Check Sample

12.2.5.1   The ICSA and ICSAB %R shall be within the control limits of ±20%
of the analyte's true value or the results shall be within ±2
times the CRQL of the analyte's true value, whichever is greater.
If the true value for a given analyte is not listed in the
certified values for the solution, then the true value shall be
assumed to be zero and the ±2 times the CRQL control limits shall
apply.

For example, for Chromium (CRQL = 2 µg/L, ICSA true value = 43
µg/L) the correct control window to use would be the greater of
±20% of the true value (0.20 x 43 µg/L = ±8.6 µg/L) or the
results shall be within ±2 times the CRQL (±4 µg/L). Therefore,
the control window for the found value for Chromium in the ICSA
is 43±8.6, or 34.4 to 51.6 µg/L.

12.2.5.2   Only if the ICS solutions are not available from the EPA, the %R
of the prepared independent Check Sample results shall be within
the control limits of ±20% of the established mean value or the
results shall be within ±2 times the analyte's CRQL of the
established mean value, whichever is greater.

12.2.6   Corrective Action for Interference Check Sample

If the deviations of the ICSA and/or ICSAB are greater than the
specified control limits, the analysis shall be terminated, the
problem corrected, the instrument recalibrated, the calibration
reverified, and reanalysis of all analytical samples. For analytes
with CRQLs less than 1000 µg/L, the ICSA and ICSAB results shall be
reported from an undiluted sample analysis.

12.3 Matrix Spike and Post-Digestion Spike Samples

12.3.1   Summary of Matrix Spike and Post-Digestion Spike Samples

The Matrix Spike sample analysis is designed to provide information
about the effect of the sample matrix on the digestion and/or
measurement methodology.

12.3.2   Frequency of Matrix Spike and Post-Digestion Spike Samples

12.3.2.1   At least one Matrix Spike sample analysis shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[1]

12.3.2.2   If a Matrix Spike sample does not meet the technical acceptance criteria listed in Section 12.3.5, a Post-Digestion Spike sample shall be performed for those analytes that do not meet the specified criteria.

12.3.3   Procedure for Matrix Spike and Post-Digestion Spike Samples

12.3.3.1   For a Matrix Spike sample, the spike is added before the digestion (i.e., prior to the addition of other reagents).

12.3.3.2   The analyte spike shall be added in the amount given in Table 6 – Spiking Levels for Matrix Spike Sample Analyses, for each element analyzed. This is the level of spike present in the final digestate.

12.3.3.3   For a Post-Digestion Spike sample, the sample that was initially used for the Matrix Spike sample analysis shall be used for the Post-Digestion Spike analysis. Spike the unspiked aliquot of the undiluted digestate at two times the indigenous level or two times the CRQL, whichever is greater.

12.3.3.4   Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for Matrix Spike sample analysis. The EPA may require that a specific sample be used for the Spike sample analysis.

12.3.4   Calculations for Matrix Spike and Post-Digestion Spike Samples

12.3.4.1   If the Matrix Spike analysis is performed on the same sample that is chosen for the Duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.4). The average of the duplicate results cannot be used for the purpose of determining %R.

12.3.4.2   Calculate the Matrix Spike and Post-Digestion Spike %R using the following equation:

EQ. 11 Matrix Spike and Post-Digestion Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,
SSR   =   Spiked Sample Result (µg/L or mg/kg) from EQ. 6 or EQ. 7
SR   =   Sample Result (original) (µg/L or mg/kg) from EQ. 6 or EQ. 7. When the sample concentration is less than the MDL, use SR=0.
SA   =   Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spiking amount used for the 'V_f' term and substituting the spiking standard concentration used for the 'C' term from EQ. 6 or EQ. 7.

NOTE: The units used for reporting SSRs will be identical to those used for reporting SRs.

[1] The EPA may require additional spike sample analyses, upon request from the EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR).

12.3.5   Technical Acceptance Criteria for Matrix Spike and Post-Digestion Spike Samples

The Matrix Spike and Post-Digestion Spike %Rs shall be within the control limits of 75-125%.

12.3.6   Corrective Action for Matrix Spike and Post-Digestion Spike Samples

12.3.6.1   If the Matrix Spike recovery is not at or within the limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with "*". An exception to this rule is granted when the sample concentration exceeds the SA concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the %R does not meet the 75-125% recovery criteria.

12.3.6.2   When the Matrix Spike recovery is outside the control limits and the sample result does not exceed four times the spike added, a Post-Digestion spike shall be performed for those analytes that do not meet the specified criteria. Follow the procedures in Section 12.3.3.

12.3.6.3   If there is more than one Matrix Spike per matrix, per SDG, if one Matrix Spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

12.4 Duplicate Sample

12.4.1   Summary of Duplicate Sample

Duplicates are analyzed to help determine sample homogeneity and laboratory precision.

12.4.2   Frequency of Duplicate Sample

One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2] Duplicates cannot be averaged for reporting.

12.4.3   Procedure for Duplicate Sample

12.4.3.1   Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. The EPA may require that a specific sample be used for duplicate sample analysis.

12.4.3.2   Prepare a second aliquot of the original sample. The duplicate sample shall be carried through the complete sample preparation procedure.

12.4.4   Calculations for Duplicate Sample

12.4.4.1   The Relative Percent Difference (RPD) for each analyte shall be calculated using the following equation:

EQ. 12 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

---

[2] The EPA may require additional duplicate sample analyses, upon request from the EPA Regional CLP COR.

```
WHERE,
S    =  Sample Result (original) (µg/L or mg/kg) from EQ. 6 or
        EQ. 7
D    =  Duplicate Sample Result (µg/L or mg/kg) from EQ. 6 or
        EQ. 7
```

12.4.5   Technical Acceptance Criteria for Duplicate Sample

12.4.5.1   The RPD shall be within the control limits of ±20 if the original and duplicate sample values are greater than or equal to five times the CRQL (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits).

12.4.5.2   The control limit shall be equal to the CRQL if either the sample or duplicate value are less than five times the CRQL or if one result is above five times the CRQL level and the other is below.

12.4.5.3   If both sample and duplicate values are less than the CRQL, the RPD is not calculated.

12.4.6   Corrective Action for Duplicate Sample

12.4.6.1   If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*".

12.4.6.2   If there is more than one duplicate sample per SDG, per matrix, and if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG.

12.5 Laboratory Control Sample

12.5.1   Summary of Laboratory Control Sample

Aqueous/water and soil/sediment Laboratory Control Samples (LCSs) shall be analyzed for each analyte using the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures employed for the samples received.

12.5.2   Frequency of Laboratory Control Sample

One LCS shall be prepared for each prepared batch of aqueous/water or soil/sediment samples in an SDG.

12.5.3   Procedure for Laboratory Control Sample

The LCS for aqueous/water and soil/sediment samples shall be prepared by spiking an aliquot of reagent water (50-100 mL for aqueous/water, 1 mL for soil/sediment) such that the final digestate shall contain each analyte at two times the CRQL for the associated matrix.

12.5.4   Calculations for Laboratory Control Sample

Calculate the results for LCS by using Equations 6 or 7 as appropriate.

EQ. 13 LCS Percent Recovery

$$\%R = \frac{Found\ (LCS)}{True\ (LCS)} \times 100$$

```
WHERE,
Found (LCS)    =   The found concentration of each analyte
                   in the LCS (µg/L or mg/kg) from EQ. 6 or
                   7.  If the analyte concentration is less
                   than the MDL, a value of zero shall be
                   substituted for the Found (LCS).
True (LCS)     =   Two times the CRQL for the appropriate
                   matrix (µg/L or mg/kg)
```

12.5.5   Technical Acceptance Criteria for Laboratory Control Sample

The %R shall be within the control limits of 70-130% for all analytes.

12.5.6   Corrective Action for Laboratory Control Sample

If the %R for the LCS for aqueous/water or soil/sediment samples are outside the control limits of 70-130%, the analyses shall be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed with appropriate new QC.

12.6 ICP-MS Serial Dilution

12.6.1   Summary of ICP-MS Serial Dilution

The Contractor shall perform Serial Dilution analyses to check for interference effects.

12.6.2   Frequency of ICP-MS Serial Dilution

The ICP-MS serial dilution analysis shall be performed on a sample from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent, prior to reporting analyte concentration data.

12.6.3   Procedure for ICP-MS Serial Dilution

12.6.3.1   Prepare an aliquot of the original sample digestate and dilute it 1:4 (five-fold) with 1% nitric acid. This dilution shall be analyzed as the serial dilution.

12.6.3.2   If the original sample requires dilution for any analyte, a 1:4 (five-fold) dilution of that dilution shall be prepared and analyzed as a serial dilution.

12.6.3.3   Samples identified as field blanks and PE samples shall not be used for serial dilution analysis.

12.6.4   Calculations for ICP-MS Serial Dilution

The percent difference for each component are calculated using the following equation:

EQ. 14 Serial Dilution Percent Difference

$$\%Difference = \frac{\left| I - S \right|}{I} \times 100$$

```
WHERE,
I      =   Initial sample result (original)
S      =   Serial dilution result.  If the analyte concentration is
           less than the MDL, a value of zero shall be substituted
           for "S".
```

12.6.5   Technical Acceptance Criteria for ICP-MS Serial Dilution

If the analyte concentration is sufficiently high (minimally a factor of 50 above the MDL in the original sample), then the serial dilution (a five-fold dilution) shall be within 10% of the original determination after correction for dilution.

12.6.6   Corrective Action for ICP-MS Serial Dilution

12.6.6.1   If the dilution analysis for one or more analytes is not within a control limit of 10%, a chemical or physical interference effect must be suspected, and the data for all affected analytes in the samples received and associated with that serial dilution must be flagged with an "*".

12.6.6.2   In the instance where there is more than one serial dilution per SDG, per matrix, if one serial dilution result is not within contract criteria, flag all the samples of the same matrix in the SDG.

12.6.6.3   If the internal standard responses for the field sample chosen for serial dilution analysis are not within the limits (identified in Section 12.7.5 – Technical Acceptance Criteria for Internal Standards), and the appropriate corrective action (two-fold dilution and reanalysis) is taken, the following shall apply to the serial dilution analysis:

- If the internal standard responses of the field sample reanalysis are within the limits, the serial dilution results are to be reported from a five-fold dilution of the reanalyzed sample.
- If the internal standard responses of the field sample reanalysis are not within the limits, the serial dilution results are to be reported from a five-fold dilution of the original sample.

12.7   Internal Standards

12.7.1   Summary of Internal Standards

Internal standardization must be used in all analyses to correct the instrument drift and physical interferences. The analyst shall monitor the responses from the internal standards and the ratios of raw uncovered responses between isotopes throughout the sample set being analyzed. This information may be used to correct potential problems caused by mass dependent drift, errors incurred in adding the internal standards, or increases in the concentrations of individual internal standards caused by background contributions from the sample.

12.7.2   Frequency of Internal Standards

Internal standards shall be present in all samples, standards, and blanks (except the tuning solution) at identical levels.

12.7.3   Procedure for Internal Standards

12.7.3.1   A minimum of five internal standards shall be used. A list of acceptable internal standards is provided in Table 5 - Internal Standards.

12.7.3.2   The internal standards selected for an analytical sequence must be consistent throughout the entire analytical sequence.

12.7.3.3   The internal standard may be added directly to an aliquot of each sample, standard, and blank, or by mixing with the sample solution prior to nebulization using a second channel of the peristaltic pump and mixing coil.

12.7.3.4   The concentration of the internal standard should be sufficiently high for good precision and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. Depending on the sensitivity of the instrument, a final concentration range of 20 µg/L to 200 µg/L of each internal standard in each digested sample, standard, and blank is recommended when the internal standards are added manually by the analyst. If the internal standards are added automatically by the instrument prior to analysis, then the manufacturer's guidelines for the appropriate concentration ranges should be followed.

12.7.3.5   If dilutions are performed on the digested samples, then the internal standards must be added after the dilution.

12.7.4   Calculation for Internal Standards

12.7.4.1   Calculate the Percent Relative Intensity using the following equation:

EQ. 15 Percent Relative Intensity

$$\%RI = \frac{I_n}{I_0} \times 100$$

WHERE,
I_n = Raw Uncorrected Intensity of the internal standard in the sample
I_0 = Raw Uncorrected Intensity of the internal standard in the calibration blank (S0)

12.7.5   Technical Acceptance Criteria for Internal Standards

The absolute response of any one internal standard must not deviate more than 60-125% from the original response in the calibration blank.

12.7.6   Corrective Action for Internal Standards

12.7.6.1   If deviations less than 60% or greater than 125% are observed in field samples, matrix spikes, or duplicate samples, the original sample shall be diluted by a factor of two, internal standards added (if not automatically added by the instrument), and the sample reanalyzed for the analyte(s) associated with the noncompliant internal standard(s).

12.7.6.2   If the internal standard responses for the diluted sample analysis are not within the limits, note this in the SDG Narrative and report the results of the undiluted original sample analysis. If the internal standard responses for the diluted sample analysis are within the limits, report the results of this analysis.

12.7.6.3   Target analyte(s) concentration(s) must be within the calibrated range before assessing internal standard response for those internal standard(s) associated with the analyte(s).

12.8   Method Detection Limit Determination

12.8.1   Before any field samples are analyzed, the MDLs shall be determined for each digestion procedure and instrument used prior to the start of contract analyses and annually thereafter. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.8.1.1   To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.8.1.2   The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.8.1.3   The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

12.9   Summary of Quality Control Operations

The QC operations performed for ICP-MS analysis are summarized in Table 7 - QC Operations for ICP-MS.

13.0   METHOD PERFORMANCE

Not applicable.

14.0   POLLUTION PREVENTION

See Section 13.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

15.0   WASTE MANAGEMENT

See Section 14.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

16.0   REFERENCES

16.1   U.S. Environmental Protection Agency, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry, Method 200.8, Revision 5.5, October 1999.

16.2   U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Method 6020B, Revision 2, July 2014.

16.3   U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. INTERFERENT AND ANALYTE CONCENTRATIONS USED FOR ICP-MS
INTERFERENCE CHECK SAMPLE (ICS)

Analytes	(µg/L)	Interferents	(µg/L)
Ag	20	Al	100000
As	20	Ca	100000
Ba	20	Fe	100000
Be	20	Mg	100000
Cd	20	K	100000
Co	20	Na	100000
Cr	40	P (as orthophosphate)	100000
Cu	25	S (as sulfate)	100000
Mn	30	C (as citrate)	200000
Ni	25	Cl	1000000
Pb	25	Mo	2000
Sb	20	Ti	2000
Se	20		
Tl	20		
V	20		
Zn	30		

NOTE: ICSA solution contains the interferents at the indicated
concentrations. The ICSA solution may be analyzed at twice the
concentration indicated when interferences are present at higher
concentrations in the sample. The ICSAB solution contains all of
the analytes and interferents listed above at the indicated
concentrations.

TABLE 2. ISOBARIC MOLECULAR-ION INTERFERENCES

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	Nd^{++}, Sm^{++}
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	SeCl, AsCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Co	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO$_2$	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	S$_2$, SO$_2$H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN			WC	

TABLE 2. ISOBARIC MOLECULAR-ION INTERFERENCES (CON'T)

Analyte	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	ScO	CaOH	TiN	MgCl	SiS	TiC	Sn^{++}
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	S_2	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	Gd^{++}
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	Sm^{++}, Gd^{++}
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		Dy^{++}, Er^{++}
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	NiOH	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	FeOH	NiN	Cl_2, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo^{++}
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	S_2	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	S_2	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	Cl_2	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, this table can be consulted if unusual samples are encountered.

TABLE 3. RECOMMENDED ISOTOPES AND MASSES FOR SELECTED ELEMENTS

Element of Interest	Analyte Masses – Choose One, or More – Calibrated	Masses to be Monitored
Aluminum	27	
Antimony	121	
Arsenic	75	77, 82 (Isobaric Equation Required), 150
Barium	135, 137	
Beryllium	9	
Cadmium	111	106, 108 (Isobaric Equation Required)
Calcium	40, 44	
Chromium	52	
Cobalt	59	
Copper	63, 65	
Iron	54, 56, 57	
Lead	206, 207, 208	
Magnesium	24, 25, 26	
Manganese	55	
Nickel	60	
Potassium	39	
Selenium	78, 82	156, 160, 164
Silver	107, 109	
Sodium	23	
Thallium	203, 205	
Vanadium	51	52, 53 (Isobaric Equation Required)
Zinc	66	
Potential Interferent		
Titanium (TiO on ^{63}Cu)		47 (No Isobaric Equation Required)
Krypton (Kr on ^{82}Se)		83 (No Isobaric Equation Required)
Molybdenum		94, 95, 96, 97, 98
Tin (Sn on ^{115}In)		118 (Isobaric Equation Required)

NOTE: Where possible, alternative isotopes are indicated. At least one of the listed masses shall be used as a quantitation ion. Those isotopes not listed shall not be used as a primary isotope for measurement, although they may be monitored for interference corrections if necessary.

TABLE 4. RECOMMENDED ELEMENTAL EXPRESSIONS FOR ISOBARIC INTERFERENCES

Element	Isobaric Correction	Expression Proportional to Elemental Concentration
As	ArCl, Se	$(1.0000)(^{75}C) - (3.127)[(^{77}C) - (0.815)(^{82}C)]$
Cd	MoO, Pd	$(1.000)(^{111}C) - (1.073)[(^{108}C) - (0.712)(^{106}C)]$
V	ClO, Cr	$(1.0000)(^{51}C) - (3.127)[(^{53}C) - (0.113)(^{52}C)]$
In	Sn	$(1.0000)(^{115}C) - (0.0140)(^{118}C)$

C – Calibration blank subtracted counts at specified mass

The coefficients in correction equations were calculated using natural isotopic abundances, and assuming zero instrumental fractionation. For each particular instrument, these coefficients must be determined experimentally using the procedures or coefficients provided by the instrument manufacturer.

The correction equations shall not be applied if appropriate interference check sample measurement demonstrates absence of interference above the CRQL.

TABLE 5. INTERNAL STANDARDS (MUST USE AT LEAST FIVE)

Internal Standard	Mass	CAS Number
Lithium	6	7439-93-2
Scandium	45	7440-20-2
Yttrium	89	7440-65-5
Rhodium	103	7440-16-6
Indium	115	7440-74-6
Terbium	159	7440-27-9
Holmium	165	7440-60-0
Lutetium	175	7439-94-3
Bismuth	209	7440-69-9

NOTE: Use of Li^6 requires enriched standard.

TABLE 6. SPIKING LEVELS FOR MATRIX SPIKE SAMPLE ANALYSIS

Analyte	Spike (µg/L)*	Spike (mg/kg)*[1]
Sb	100	10
As	40	4
Ba	2000	200
Be	50	5
Cd	50	5
Cr	200	20
Co	500	50
Cu	250	25
Pb	20	2
Mn	500	50
Ni	500	50
Se	100	10
Ag	50	5
Tl	50	5
V	500	50
Zn	500	50

*Level in the final prepared sample

[1] Concentrations in the spike sample when the dry weight of 1 gram of sample is taken for analysis. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values.

EQ. 16 Spiking Level Adjustment

$$mg/kg = µg/L \times \frac{final\ volume\ (L)}{sample\ weight\ (g)}$$

TABLE 7. QC OPERATION FOR ICP-MS

QC Operation	Frequency
ICP-MS Tune	Prior to calibration.
Instrument Calibration	Each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration for each mass used.
Continuing Calibration Verification	For each mass used, at a frequency of every 2 hours and at the beginning and end of each analytical sequence.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Blank	Every 2 hours and at the beginning and end of each analytical sequence. Performed immediately after the CCV.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Interference Check Sample	At the beginning of each analytical sequence after the ICB but before the CCV.
Matrix Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Digestion Spike	Each time Matrix Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Laboratory Control Sample	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Serial Dilution for ICP	For each matrix type or for each SDG, whichever is more frequent.
Method Detection Limit Determination	Prior to start of contract analyses, annually thereafter, and after major instrument adjustment.

EXHIBIT D

COLD VAPOR MERCURY ANALYSIS

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Exhibit D – Cold Vapor Mercury Analysis

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Exhibit D – Cold Vapor Mercury Analysis

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1.0   SCOPE AND APPLICATION

This exhibit provides procedures for the use of a cold vapor technique
with Atomic Absorption (AA) to determine total mercury in
aqueous/water, leachate derived from the Toxicity Characteristic
Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure
(SPLP), and soil/sediment samples taken from hazardous waste sites.

In addition to inorganic forms of mercury, organic mercury may also be
present. These organo-mercury compounds will not respond to the cold
vapor AA technique unless they are first broken down and converted to
mercuric ions. Potassium permanganate oxidizes many of these
compounds, but studies have shown that a number of organo-mercury
compounds, including phenyl mercuric acetate and methyl mercuric
chloride, are only partially oxidized by this reagent. Potassium
persulfate has been found to give approximately 100% recovery when used
as the oxidant with these compounds. Therefore, a persulfate oxidation
step following the addition of the permanganate has been included in
most preparation procedures to ensure that organo-mercury compounds, if
present, will be oxidized to the mercuric ion before measurement. A
heat step is required for methyl mercuric chloride when present in, or
spiked to, a natural system.

2.0   SUMMARY OF METHOD

2.1   General Method Overview

This method is based on the absorption of radiation at 253.7 nanometers
(nm) by mercury vapor. Inorganic and some organic forms of mercury are
chemically reduced to the free atomic state by reacting the sample with
a strong reducing agent like stannous chloride or stannous sulfate in a
closed reaction vessel. The volatile free mercury is then driven from
the reaction flask by bubbling air through the solution. Mercury atoms
are carried in the air stream through tubing connected to an absorption
cell, which is placed in the light path of the AA spectrophotometer.
Sometimes the cell is heated slightly to avoid water condensation. As
the mercury atoms pass into the sampling cell, measured absorbance
rises indicating the increasing concentration of mercury atoms in the
light path. Some systems allow the mercury vapor to pass from the
absorption tube to waste, in which case the absorption peaks and then
falls as the mercury is depleted. The highest absorbance observed
during the measurement or the associated peak area are usually taken as
the analytical signal.

2.2   Summary of Preparation and Analysis Procedures

2.2.1   Heated Acid Digestion and Analysis of Aqueous/Water and TCLP/SPLP
         Leachate Samples (based on the EPA Method 7470A)

2.2.2   Heated Acid Digestion and Analysis of Soil/Sediment Samples (based
         on the EPA Method 7471B)

3.0   DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0   INTERFERENCES

4.1   Chlorides

Samples high in chlorides have shown a positive interference, and require additional potassium permanganate [as much as 25 milliliters (mL)]. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253.7 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

4.2   Sulfides

Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 milligrams/Liter (mg/L) of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

4.3   Copper

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on the recovery of mercury from spiked samples.

4.4   Oxidizable Organic Materials

Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized by this procedure. When this occurs, the recovery of organic mercury will be low. The problem can be eliminated by reducing the amount of the original sample or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

5.0   SAFETY

See Section 12.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

6.0   EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1   Glassware/Labware

6.1.1   Graduated cylinders

6.1.2   Various volumetric flasks (Class A) and calibrated pipets. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.1.3   Suitable digestion vessels (300 mL BOD bottles, hot block digestion tubes, etc.), along with a suitable heating source/water bath for heating of the samples to about 95°C

6.1.4   Balances – Top loader balance, 300 gram (g) capacity, and a minimum sensitivity of ±1.0 mg

A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each balance and be accurate to ±1.0 mg. The masses that are used to check the balances daily must be checked on a monthly basis using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '1' or '2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified every five years or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates that these criteria have been met.

6.2   Cold Vapor Atomic Absorption Spectrometer

Consisting of an AA spectrometer equipped with a flow-through absorption cell and a mercury hollow cathode lamp or other suitable light source. The analysis system shall also include: a manifold/pump system for mixing reagents with previously digested samples, a liquid-vapor separator, and a vapor dryer. The spectrometer shall have or be linked to a suitable computer system for data processing.

AA Spectrophotometer – Any AA unit having an open sample presentation area in which to mount the absorption cell would be suitable. Instrument settings recommended by the particular manufacturer should be followed. The instrument must be capable of meeting the specified Contract Required Quantitation Limits (CRQLs) for mercury.

7.0   REAGENTS AND STANDARDS

7.1   Reagents

7.1.1   Reagent water - The purity of this water must be equivalent to ASTM Type II water (ASTM D1193-06). Use this water for all reagents, standards, and dilutions.

7.1.2   Sulfuric acid - Concentrated 95-98% - Reagent grade.

7.1.3   Sulfuric acid, 0.5N - Dilute 14.0 mL of concentrated sulfuric acid to 1 L.

7.1.4   Hydrochloric acid - Concentrated 32-38%. Reagent grade of low mercury content.

7.1.5   Nitric acid - Concentrated 67-70% - Reagent grade of low mercury content. It may be necessary to distill the nitric acid if impurities are detected in blanks.

7.1.6   Aqua regia - Prepare immediately prior to use. Carefully add three volumes of concentrated hydrochloric acid to one volume of concentrated nitric acid.

7.1.7   Sodium chloride-hydroxylamine sulfate solution, 12% solution (w/v) - Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate in reagent water and dilute to 100 mL.

   NOTE: Hydroxylamine hydrochloride may be used in place of hydroxylamine sulfate.

7.1.8   Potassium permanganate ($KMnO_4$) - 5% solution (w/v). Dissolve 5 g of potassium permanganate in 100 mL of reagent water.

7.1.9   Potassium persulfate - 5% solution (w/v). Dissolve 5 g of potassium persulfate in 100 mL of reagent water.

7.1.10   Stannous sulfate, (10% w/v) - Dissolve 25 g stannous sulfate to 250 mL of 0.5N sulfuric acid. This mixture is a suspension and should be stirred continuously during use.

   NOTE: Stannous chloride may be used in place of stannous sulfate.

7.2   Standards

7.2.1   Introduction

   The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit D - Introduction to Inorganic Analytical Methods, Section 11.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

7.2.2   Stock Standard Solutions

   Stock standard solutions may be purchased from a reputable source or prepared from reagent grade chemicals or metals (at least 99.99% pure).

   CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.

7.2.2.1   Stock mercury solution - Dissolve 0.1354 g of mercuric chloride in 75 mL of reagent water. Add 10 mL of concentrated nitric acid and adjust the volume to 100.0 mL [1.0 mL = 1.0 mg Hg].

7.2.3   Working Standards

7.2.3.1   Working Mercury Solution

Make successive dilutions of the stock mercury solution (see Section 7.2.2) to obtain a working standard containing 0.1 micrograms/milliliter (µg/mL). This working standard and the dilutions of the stock mercury solution shall be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. Acid should be added to the flask as needed before the addition of the aliquot. From this solution, prepare calibration standards. Standards must be prepared with samples (See Section 10.0).

7.2.4   Initial Calibration Verification Solution

7.2.4.1   The Initial Calibration Verification (ICV) solution shall be obtained from the EPA.

7.2.4.1.1   If the solution is not available from the EPA, then the ICV solution shall be prepared by the laboratory using a certified solution from an independent source. An independent source is defined as a standard from a different source than that used in the standards for instrument calibration. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range.

7.2.4.1.2   The ICV standard shall be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier.

7.3   Blanks

Two types of blanks are required for this method. A Calibration Blank is used to establish the analytical calibration curve and the Preparation Blank (see Section 12.1) is used to assess possible contamination from the sample preparation procedure and to assess spectral background.

7.3.1   Calibration Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Calibration Blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are identical in composition to the Calibration Blank and are used to monitor for analyte carryover.

7.3.2   Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Preparation Blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Soil/sediment blanks shall use 0.5-0.6 mL of reagent water.

8.0   SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1   Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in glass or polyethylene containers. Aqueous/water samples must be preserved with nitric acid to pH less than or equal to 2 immediately after collection. All soil/sediment samples must be iced or refrigerated at ≤6°C but not frozen from the time of collection until receipt at the laboratory.

8.2   Sample Storage

All aqueous/water and soil/sediment samples must be stored at ≤6°C but not frozen from the time of sample receipt until digestion.

8.2.1   Unused Sample Storage

The Contractor shall retain the unused portion of the aqueous/water and soil/sediment samples for a period of 60 days after delivery of a complete, reconciled data package to the EPA. The unused portion may be stored at room temperature.

8.2.2   Digestate Sample Storage

Digestions shall not be retained. Any reanalyses of the sample shall be performed using a freshly digested aliquot of the sample.

8.2.3   Container Storage

The Contractor shall retain the empty sample containers for 60 days after the delivery of a complete reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.3   Contract Required Holding Time

The holding time for mercury is 26 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of TCLP or SPLP leachates is 26 days from the date of extraction.

9.0   CALIBRATION AND STANDARDIZATION

9.1   Instrument Operating Parameters

Because of the differences between various manufacturers and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL) and precision must be investigated and established for mercury on that particular instrument. All measurements must be within the instrument operating range. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used to satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2   Pre-Calibration Routine

Prior to calibration, the Contractor shall set up each instrument with the operating parameters established in Section 9.1 and conduct any necessary adjustments. The instrument must be allowed to become stable (usually 30 minutes) before calibration is performed.

9.3   Instrument Calibration Procedure

9.3.1   Summary of Instrument Calibration

Prior to sample analysis, the Contractor shall calibrate each instrument to determine sensitivity and linearity of the response.

9.3.2   Frequency of Instrument Calibration

Each instrument shall be calibrated daily or once every 24 hours, each time the instrument is set up, or after ICV, ICB, Continuing Calibration Verification (CCV), or CCB failure. The instrument calibration date and time shall be included in the raw data.

9.3.3   Procedure for Instrument Calibration

9.3.3.1   Each instrument shall be calibrated according to the manufacturer's recommended procedures.

9.3.3.2   At least six calibration standards shall be used. The calibration standards shall be prepared as in Section 7.2.3.1. One of the standards shall be a blank standard and one shall be at or below the CRQL, but greater than the MDL. The rest of the standards shall be uniformly spread out in graduated amounts over the appropriate calibration range (typical range is 0.20 to 10.0 µg/L).

9.3.4   Calculations for Instrument Calibration

9.3.4.1   The calibration curve shall be calculated using linear regression by plotting the concentration of the standard (in µg/L) on the X-axis versus the instrument response on the Y-axis. The instrument response is the measured absorbance (displayed as a peak area or height) for each standard.

9.3.4.2   The Contractor may use standard linear regression, weighted linear regression (1/concentration, 1/square of concentration, variance, 1/variance, standard deviation, 1/standard deviation, or 1/square of standard deviation), or linear regression with zero force calibration models, as appropriate, for the above calculation. No other types of equations (e.g., quadratic) are to be used.

9.3.4.3   The calibration curve shall be checked to establish the representativeness of the data that were used to produce it. This check involves re-fitting the data of the non-blank calibration standards back to the calibration curve, and computing the difference between the calculated concentration and the expected concentration for each of these standards. The difference is then divided by the expected concentration of the respective standard and multiplied by 100.

9.3.5   Technical Acceptance Criteria for Instrument Calibration

9.3.5.1   The correlation coefficient of the calibration curve shall be greater than or equal to 0.995.

9.3.5.2   The Percent Difference (%D) for each of the standards shall be within the control limits of ±30%.

9.3.5.3   If a standard is analyzed at a concentration that is below the CRQL and the %D criteria is not met, that standard can be excluded from the calibration curve as long as the lowest non-zero standard is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.3.6   Corrective Action for Instrument Calibration

9.3.6.1   Sample analysis shall not begin until the criteria described in Section 9.3.5 have been met.

9.3.6.2   Any changes or corrections to the analytical system shall be followed by recalibration.

9.4   Initial Calibration Verification

9.4.1   Summary of Initial Calibration Verification

Prior to sample analysis, the Contractor shall demonstrate the accuracy of the instrument calibration for each instrument through the analysis of an ICV standard.

9.4.2   Frequency of Initial Calibration Verification

The ICV shall be analyzed immediately after the instrument has been calibrated.

9.4.3   Procedure for Initial Calibration Verification

9.4.3.1   The ICV shall be analyzed at the wavelength used to report final results.

9.4.3.2   The ICV shall reflect the conditions of analysis of the associated analytical samples.

9.4.4   Calculations for Initial Calibration Verification

The Percent Recovery (%R) of the ICV shall be calculated using the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{Found\ (ICV)}{True\ (ICV)} \times 100$$

WHERE,
Found (ICV)   = The found concentration of mercury in the ICV Solution

True (ICV)   = The expected concentration of mercury in the ICV Solution

9.4.5   Technical Acceptance Criteria for Initial Calibration Verification

The ICV %R shall be within the control limits of 85-115%.

9.4.6   Corrective Action for Initial Calibration Verification

If the recovery is outside the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

9.5   Continuing Calibration Verification

9.5.1   Summary of Continuing Calibration Verification

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration through the analysis of a CCV standard. The Contractor shall not analyze a blank immediately prior to the analysis of the CCV.

9.5.2   Frequency of Continuing Calibration Verification

9.5.2.1   A CCV shall be analyzed at the beginning of the analytical sequence and after the last analytical sample. A CCV shall also be analyzed at a frequency not to exceed 1 hour during an analytical sequence. See the example analytical sequence in Section 10.3.3.

9.5.2.2   The analytical sequence can continue for 24 hours as long as samples are being continuously analyzed without the instrument being turned off and successive CCV standards meet the technical acceptance criteria in Section 9.5.5.

9.5.3   Procedure for Continuing Calibration Verification

9.5.3.1   The CCV standard shall be prepared using the same source and in the same acid matrix as the calibration standards at or near the mid-level of the calibration curve. The CCV shall be prepared according to Section 10.0.

9.5.3.2   The same CCV standard shall be used throughout the analytical sequences for an SDG.

9.5.3.3   The CCV shall be analyzed at the wavelength used to report final results.

9.5.3.4   Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5.4   Calculations for Continuing Calibration Verification

9.5.4.1   The %R of the CCV shall be calculated using the following equation:

EQ. 2 CCV Percent Recovery

$$\%R = \frac{\text{Found (CCV)}}{\text{True (CCV)}} \times 100$$

WHERE,
Found (CCV)   =   The found concentration of mercury in the CCV Solution

True (CCV)   =   The expected concentration of mercury in the CCV Solution

9.5.5   Technical Acceptance Criteria for Continuing Calibration Verification

9.5.5.1   The CCV %R shall be within the control limits of 85-115%.

9.5.5.2   All samples shall be analyzed within 1 hour of an acceptable opening and closing CCV.

9.5.6   Corrective Action for Continuing Calibration Verification

If the deviations of the CCV are greater than the specified control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all analytical samples analyzed since the last compliant calibration verification shall be performed.

9.6   Initial and Continuing Calibration Blank

9.6.1   Summary of Calibration Blank

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration baseline through the analysis of an ICB and CCB.

9.6.2   Frequency of Calibration Blank

9.6.2.1   An ICB shall be analyzed immediately after the instrument has been calibrated and the calibration verified by the analysis of the ICV.

9.6.2.2   Each CCB shall be analyzed immediately after the analysis of a CCV.

9.6.3   Procedure for Calibration Blank

9.6.3.1   The ICB and CCB samples shall be analyzed at the wavelength used for reporting final results.

9.6.3.2   The ICB analyzed shall reflect the conditions of analysis of the associated analytical samples.

9.6.3.3   Each CCB analyzed shall reflect the conditions of analysis of the associated analytical samples (the preceding analytical samples up to the previous CCB as applicable and the analytical samples following this CCB up to the next CCB as applicable).

9.6.4   Calculations for Calibration Blank

The results for the ICB and CCB samples shall be calculated using Equation 3 in Section 11.0.

9.6.5   Technical Acceptance Criteria for Calibration Blank

The absolute value of each calibration blank result shall be less than or equal to the CRQL for aqueous/water samples.

9.6.6   Corrective Action for Calibration Blank

If the absolute value of the calibration blank exceeds the CRQL for aqueous/water samples, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all affected analytical samples analyzed since the last compliant calibration blank performed.

10.0 PROCEDURE

10.1 Aqueous/Water Sample Preparation

10.1.1 If the sample pH was ≤2 at the time of sample receipt, the Contractor shall proceed to Section 10.1.2.

If the sample pH was adjusted at the time of sample receipt (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1), the Contractor shall take a second pH measurement, prior to removing an aliquot of the sample for digestion, to verify that the sample was properly preserved upon receipt. If the second pH measurement is ≤2, proceed to Section 10.1.2. If the second pH measurement is >2, the Contractor shall add sufficient nitric acid to the sample to reduce the pH to ≤2, return the sample to storage for a minimum of 16 hours before proceeding with the preparation of the sample, and document the pH adjustment in the SDG Narrative.

10.1.2 For preparation of the standards, transfer aliquots of the working mercury solution (see Section 7.2.3.1) to a series of suitable digestion vessels. Add enough reagent water to each digestion vessel to make a total volume of 100 mL (±1.0 mL) and mix thoroughly. The acidity of all of the standards should be maintained at 0.15% nitric acid. This acid should be added to the digestion vessel as needed prior to the dilution of the working standard. Standards must be prepared fresh daily.

10.1.3 For preparation of the samples, shake the sample until well mixed and transfer an aliquot of 100 mL (±1.0 mL), containing not more than 1.0 µg of mercury, to a suitable digestion vessel.

10.1.4 Add 5 mL of concentrated sulfuric acid and 2.5 mL of concentrated nitric acid to each of the digestion vessels, mixing after each addition.

10.1.5 Add 15 mL of 5% potassium permanganate solution to each digestion vessel. Some samples (e.g., sewage samples) may require additional potassium permanganate. Shake and add additional portions of potassium permanganate solution, if necessary, until the purple color persists for at least 15 minutes. Ensure that equal amounts of potassium permanganate solution are added to all standards, blanks, and samples.

10.1.6 Add 8 mL of 5% potassium persulfate solution to each digestion vessel and heat for 2 hours in a water bath or block digester maintained at 95°C (±3°C). Allow to cool.

10.1.7 Add 6 mL of 12% sodium chloride-hydroxylamine sulfate solution to reduce the excess potassium permanganate.

10.1.8 All standards and samples must be at the same final volume. Reagent water can be used to make any necessary final volume adjustments.

10.1.9 After digestion, the standards and samples may stand at room temperature for up to 48 hours prior to analysis. However, it is recommended that the digested standards and samples be analyzed as soon as possible. Proceed to Section 10.3 for analysis.

10.1.10 A reduced volume of 50 mL can be used for all standards, blanks, and samples for this digestion procedure. If the reduced volume is used, all standards and reagents used in the digestion process shall be reduced by half of their original required amounts.

10.2 Soil/Sediment Sample Preparation

10.2.1 For preparation of the standards, transfer aliquots of the working mercury solution (see Section 7.2.3.1) to a series of suitable digestion vessels. Add enough reagent water to each digestion vessel to make a total volume of 10 mL (±0.1 mL) and mix thoroughly. The acidity of all of the standards should be maintained at 0.15% nitric acid. This acid should be added to the digestion vessel as needed prior to the dilution of the working standard. Standards must be prepared fresh daily.

10.2.2 For preparation of the samples, mix the sample thoroughly to achieve homogeneity. Weigh (to the nearest 0.01 g) an aliquot amount of 0.50-0.60 g and place in the bottom of a suitable digestion vessel. Add 5 mL of reagent water to each sample.

10.2.3 Add 5 mL of aqua regia to each of the digestion vessels and heat for 2 minutes at 95°C (±3°C) in a water bath or block digester. Allow the contents of each digestion vessel to cool.

10.2.4 Add 50 mL of reagent water and 15 mL of 5% potassium permanganate solution. Mix thoroughly and heat again for 30 minutes at 95°C (±3°C) in a water bath or block digester. Allow to cool.

10.2.5 Add 6 mL of 12% sodium chloride-hydroxylamine solution to each digestion vessel to reduce the excess potassium permanganate.

CAUTION: This addition should be performed under a hood, as chlorine could be evolved.

10.2.6 Add 55 mL of reagent water to each sample or 50 mL of reagent water to each standard. All standards and samples must be at the same final volume. Reagent water can be used to make any final volume adjustments, if necessary.

10.2.7 After digestion, the standards and samples may stand at room temperature for up to 48 hours prior to analysis. However, it is recommended that the digested standards and samples be analyzed as soon as possible. Refer to Section 10.3 for analysis.

10.3 Sample Analysis

10.3.1 Set up the automated analyzer using the recommendations as provided by the manufacturer. Set up the manifold and fill the reagent reservoir with the 10% (w/v) stannous sulfate solution (prepared in 0.5 N sulfuric acid). All reagent and sample lines should be cleaned according to the manufacturer's recommendations.

10.3.2 Transfer appropriate aliquots of the digested standards and samples to the autosampler in the order as suggested by the manufacturer.

10.3.3   Example Analytical Sequence for Mercury Including the Instrument
         Calibration:

```
S##
S##
S##
S##
S##
S##
ICV
ICB
CCV###
CCB###
Samples
CCV###
CCB###
samples
CCV###
CCB###, etc.
```

10.3.4   Complete the analysis of all of the digested standards and samples
         and construct the calibration curve. The calibration curve shall be
         constructed based on the concentration of mercury (in µg/L) in the
         undigested standards, ignoring the volume of reagents added during
         the digestion process.

10.3.5   If a sample's response exceeds the calibrated range of the
         instrument, the laboratory shall dilute the sample and reanalyze.
         Dilute a portion of the previously digested sample, which has not
         been treated with stannous sulfate, using a solution which maintains
         the same acid and other reagent concentrations as are present in the
         calibration standards (e.g., one of the calibration blanks). The
         laboratory shall then promptly analyze the diluted sample.

10.3.6   After the analysis is complete, clean out the system and all of the
         reagent and sample lines according to the manufacturer's
         recommendations.

11.0   DATA ANALYSIS AND CALCULATIONS

Calculate the mercury concentration using the following equations.

11.1   Aqueous/Water and TCLP/SPLP Leachate Sample Calculation

EQ. 3   Aqueous/Water and TCLP/SPLP Leachate Sample Concentration

$$\text{Hg Concentration} (\mu g/L) = C \times DF$$

WHERE,
C   =   Instrument value in µg/L from the calibration curve
DF =   Dilution Factor

NOTE: Convert units to mg/L for TCLP leachates by dividing the final
      calculated concentration by 1000.

11.2   Soil/Sediment Sample Calculation

EQ. 4   Soil/Sediment Sample Concentration

$$\text{Hg Concentration} (\text{mg/kg dry weight}) = C \times \frac{1}{W \times S} \times DF \times 0.1$$

WHERE,
C   =   Instrument value in µg/L from the calibration curve
W   =   Initial aliquot amount (g)
S   =   % Solids/100 (Exhibit D – General Inorganic Analysis,
        Section 10.1.1)
DF =   Dilution Factor

11.3   Adjusted Contract Required Quantitation Limit Calculation

11.3.1   Calculate the adjusted CRQL for aqueous/water or TCLP/SPLP leachate
         samples, by multiplying the CRQL (µg/L) by the dilution factor.
         Convert units to mg/L for TCLP leachate samples.

11.3.2   Calculate the adjusted CRQL for soil/sediment samples using the
         following equation:

EQ. 5   Adjusted Soil/Sediment CRQL

$$\text{Adjusted CRQL} (\text{mg/kg}) = C \times \frac{W_m}{W \times S} \times DF$$

WHERE,
C   =   CRQL (mg/kg)
W_m =   Method required minimum sample weight (g) (0.50 g)
W   =   Initial aliquot amount (g)
S   =   % Solids/100 (Exhibit D – General Inorganic Analysis,
         Section 10.1.1)
DF   =   Dilution Factor

12.0 QUALITY CONTROL

12.1 Preparation Blank Sample

12.1.1   Summary of Preparation Blank Sample

The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.1.2   Frequency of Preparation Blank Sample

At least one Preparation Blank shall be prepared with every SDG, or with each preparation batch, whichever is more frequent.

12.1.3   Procedure for Preparation Blank Sample

The Preparation Blank shall be carried through the complete sample preparation procedure for the matrix and contain the same acid concentration in the final digestate as the samples.

12.1.4   Calculations for Preparation Blank Sample

Calculate the results for aqueous Preparation Blanks by using Equation 3. Calculate the results for soil/sediment Preparation Blanks by using Equation 4.

12.1.5   Technical Acceptance Criteria for Preparation Blank Sample

12.1.5.1   The absolute value of the Preparation Blank result shall be less than or equal to the CRQL.

12.1.5.2   The mercury concentration in the Preparation Blank may be greater than the CRQL, if the concentration of mercury in the associated samples is greater than or equal to 10 times the blank concentration.

12.1.5.3   The mercury concentration in the Preparation Blank may be less than the negative CRQL if the concentration in the associated samples is greater than or equal to 10 times the CRQL.

12.1.6   Corrective Action for Preparation Blank Sample

12.1.6.1   If the mercury concentration in the Preparation Blank is greater than the CRQL, and the concentration of mercury in any of the associated samples is less than 10 times the blank concentration, then all samples with less than 10 times the blank concentration shall be reprepared and reanalyzed with appropriate new QC.

12.1.6.2   If the mercury concentration in the Preparation Blank is less than the negative CRQL and the concentration in the associated samples is less than 10 times the CRQL, then all samples with less than 10 times the CRQL concentration shall be reprepared and reanalyzed with appropriate new QC.

12.2 Matrix Spike Sample

12.2.1   Summary of Matrix Spike Sample

The Matrix Spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and/or measurement methodology.

12.2.2   Frequency of Matrix Spike Sample

At least one Matrix Spike sample analysis shall be performed on each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[1]

12.2.3   Procedure for Matrix Spike Sample

12.2.3.1   The spike is added before the digestion (i.e., prior to the addition of other reagents).

12.2.3.2   The analyte spike shall be added at 1 µg/L for aqueous/water and leachate samples, or at 0.5 milligrams/kilogram (mg/kg) for soil/sediment samples. Adjustment shall be made to maintain these spiking levels when the weight of sample taken deviates by more than 10% of these values. This is the level of spike present in the final digestate.

12.2.3.3   Samples identified as field blanks and Performance Evaluation (PE) samples shall not be used for Matrix Spike sample analysis. The EPA may require that a specific sample be used for the Matrix Spike sample analysis.

12.2.4   Calculations for Matrix Spike Sample

12.2.4.1   If the Matrix Spike analysis is performed on the same sample that is chosen for the Duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.3). The average of the duplicate results cannot be used for the purpose of determining %R.

12.2.4.2   Calculate the Matrix Spike %R using the following equation:

EQ. 6 Matrix Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,
SSR   =   Spiked Sample Result (µg/L or mg/kg) from EQ. 3 or EQ. 4
SR   =   Sample Result (original) (µg/L or mg/kg) from EQ. 3 or EQ. 4. When the sample concentration is less than the MDL, use SR=0.
SA   =   Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by substituting the spike concentration specified in Section 12.2.3.2 for the 'C' term from EQ. 3 or EQ.4.

NOTE: The units used for reporting SSRs will be identical to those used for reporting SRs.

12.2.5   Technical Acceptance Criteria for Matrix Spike Sample

The Matrix Spike %R shall be within the control limits of 75-125%.

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[1] The EPA may require additional spike sample analyses, upon request from the EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR).

12.2.6   Corrective Action for Matrix Spike Sample

12.2.6.1   If the Matrix Spike recovery is not within the control limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with "*". An exception to this rule is granted when the sample concentration exceeds the SA concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the %R does not meet the 75-125% recovery criteria.

12.2.6.2   If there is more than one Matrix Spike per matrix, per SDG, if one Matrix Spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

12.3   Duplicate Sample

12.3.1   Summary of Duplicate Sample

Duplicates are analyzed to help determine sample homogeneity and laboratory precision.

12.3.2   Frequency of Duplicate Sample

12.3.2.1   One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2]

12.3.2.2   Duplicate sample analyses cannot be averaged for reporting.

12.3.3   Procedure for Duplicate Sample

12.3.3.1   Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. The EPA may require that a specific sample be used for duplicate sample analysis.

12.3.3.2   Prepare a second aliquot of the original sample. The duplicate sample shall be carried through the entire sample preparation procedure.

12.3.4   Calculations for Duplicate Sample

12.3.4.1   The Relative Percent Difference (RPD) for each analyte shall be calculated using the following equation:

EQ. 7 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S - D|}{(S+D)/2} \times 100$$

WHERE,
S   =   Sample Result (original) (µg/L or mg/kg) from EQ. 3 or EQ. 4
D   =   Duplicate Sample Result (µg/L or mg/kg) from EQ. 3 or EQ. 4

12.3.5   Technical Acceptance Criteria for Duplicate Sample

12.3.5.1   The RPD shall be within the control limits of ±20 if the original and duplicate sample values are greater than or equal to five times the CRQL (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits).

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[2] The EPA may require additional duplicate sample analyses, upon request from the EPA Regional CLP COR.

12.3.5.2   The control limit shall be equal to the CRQL if either the sample or duplicate value are less than five times the CRQL or if one result is above five times the CRQL level and the other is below.

12.3.5.3   If both sample and duplicate values are less than the CRQL, the RPD is not calculated.

12.3.6   Corrective Action for Duplicate Sample

12.3.6.1   If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*".

12.3.6.2   If there is more than one duplicate sample per matrix, per SDG, and if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG.

12.4 Method Detection Limit Determination

12.4.1   Before any field samples are analyzed, the MDLs shall be determined for each digestion procedure and instrument used prior to the start of contract analyses and annually thereafter. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.4.1.1   To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.1.2   The Contractor shall prepare the MDL samples by each digestion procedure used and shall analyze these samples on each instrument used.

12.4.1.3   The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

12.5 Summary of Quality Control Operations

The quality control (QC) operations performed for mercury analysis are summarized in Table 1 – QC Operations for Mercury Analysis.

13.0   METHOD PERFORMANCE

Not applicable.

14.0   POLLUTION PREVENTION

See Section 13.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

15.0   WASTE MANAGEMENT

See Section 14.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

16.0   REFERENCES

16.1 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Method 7470A, Revision 1, September 1994.

16.2 U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste (SW-846), Method 7471B, Revision 2, February 2007.

16.3 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

17.0   TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. QC OPERATIONS FOR MERCURY ANALYSIS

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment
Initial Calibration Verification	Following each instrument calibration.
Continuing Calibration Verification	At a frequency of every hour of an analytical sequence, and at the beginning and end of each analytical sequence.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Blank	Every hour and at the beginning and end of each analytical sequence. Performed immediately after the CCV.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Matrix Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Method Detection Limit Determination	Prior to start of contract analyses, annually thereafter, and after major instrument adjustment.

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EXHIBIT D

TOTAL CYANIDE ANALYSIS

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Exhibit D – Total Cyanide Analysis

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Exhibit D – Total Cyanide Analysis

Table of Contents

1.0   SCOPE AND APPLICATION

This exhibit provides procedures to determine the concentration of total cyanide in aqueous/water, leachate derived from the Synthetic Precipitation Leaching Procedure (SPLP), and soil/sediment samples taken from hazardous waste sites.

2.0   SUMMARY OF METHOD

2.1   General Method Overview

This method describes cyanide determination by spectrophotometry. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation, using either a midi- or micro-distillation process, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined spectrophotometrically.

In the semiautomated spectrophotometric measurement, the cyanide is converted to cyanogen chloride (CNCl), by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read between 570 and 580 nanometers (nm). To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

2.2   Summary of Distillation Procedures

2.2.1   Midi-Distillation of Aqueous/Water, SPLP Leachate, and Soil/Sediment Samples [based on EPA Method 335.4 (Rev. 1, 1993) and SM 4500-CN E (approved 1999)]

2.2.2   Micro-Distillation of Aqueous/Water, SPLP Leachate, and Soil/Sediment Samples [based on Lachat QuikChem Method 10-204-00-1-X (approved by EPA 3/12/2007)]

3.0   DEFINITIONS

See Exhibit G - Glossary of Terms for a complete list of definitions.

4.0   INTERFERENCES

Several interferences may contribute to inaccuracies in the determination of cyanide in aqueous/water, SPLP leachate, and soil/sediment samples by spectrophotometry. Some of the known interferences are aldehydes, nitrate-nitrite, oxidizing agents such as chlorine, thiocyanate, thiosulfate, and sulfide. Some interferences are eliminated or reduced by using the distillation procedure. Some specific interferences that are commonly encountered are further discussed in Sections 4.1 through 4.4.

4.1   Sulfides

Sulfides adversely affect the spectrophotometric procedure. The sample shall be tested for the presence of sulfides as described in Section 10.1.2.

4.2   Surfactants

The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 anti-foam agent, or equivalent, will prevent the foam from collecting in the condenser.

4.3   Oxidizing Agents

Oxidizing agents such as chlorine decompose most of the cyanides. The sample shall be tested for the presence of oxidizing agents as described in Section 10.1.2.

4.4   Nitrates-Nitrites

High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid that will react with some organic compounds to form oximes. These oximes will decompose under test conditions to generate HCN. The samples shall be tested for presence of nitrate and nitrite as described in Section 10.3.1.5.

5.0   SAFETY

See Section 12.0 in Exhibit D – Introduction to Inorganic Analytical Methods.

6.0   EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here; however, a demonstration of equivalent performance meeting the requirements of this Statement of Work (SOW) is the responsibility of the Contractor. The Contractor shall document any use of alternate equipment or supplies in the Sample Delivery Group (SDG) Narrative.

6.1   Glassware/Labware

6.1.1   Assorted volumetric glassware (Class A), and calibrated pipettes and micropipettes. Manufacturer's instructions should be followed for the calibration and maintenance of adjustable pipettes.

6.1.2   Balances – Top loader balance, 300 gram (g) capacity, and a minimum sensitivity of ±1.0 milligram (mg)

A balance calibration must be checked with known masses once per each day of use. This verification consists of a check with two weights covering the range expected (approximately ±50% of the expected measured mass) for each balance and be accurate to ±1.0 mg. The masses that are used to check the balances daily must be checked on a monthly basis using National Institute of Standards and Technology (NIST)-traceable known reference masses (Class '1' or '2') as defined by ASTM E617-97 (2008) or equivalent (e.g., earlier Class 'S' defined masses). All balances must be checked at least once annually by a certified technician. The reference masses used by the Contractor must be recertified every five years or sooner if there is reason to believe damage (corrosion, nicks) has occurred. The Contractor shall maintain documentation that demonstrates that these criteria have been met.

6.2   Distillation Apparatus

6.2.1   Midi-Distillation Apparatus

6.2.1.1   Midi-reflux distillation apparatus

6.2.1.2   Heating block - Capable of maintaining 125°C (±5°C)

6.2.2   Micro-Distillation Apparatus

6.2.2.1   Heating block - capable of maintaining 120°C (±5°C)

6.2.2.2   Micro-distillation tubes - Sample tubes and Collector tubes,
            either pre-filled or user-filled with trapping solution

6.2.2.3   Tube press

6.3   Flow Injection Analyzer with accessories

6.3.1   Sampler

6.3.2   Pump

6.3.3   Cyanide cartridge

6.3.4   Spectrophotometer with 50 millimeter (mm) flow cells and 580 nm
          filter

6.3.5   Chart recorder or data system

7.0   REAGENTS AND STANDARDS

7.1   Reagents

7.1.1   Reagent water - The purity of this water must be equivalent to ASTM
          Type II water (ASTM D1193-06). Use this water for all reagents,
          standards, and dilutions.

7.1.2   Lead Acetate Test Paper.

7.1.3   Cadmium carbonate (Powdered).

7.1.4   Potassium Iodide – Starch Test Paper.

7.1.5   Ascorbic acid (Crystals).

7.1.6   Midi-Distillation Reagents

7.1.6.1   Sodium hydroxide solution, 0.25N - Dissolve 10.0 g sodium
            hydroxide in reagent water and dilute to 1 Liter (L).

7.1.6.2   Sodium hydroxide solution, 1.25N - Dissolve 50.0 g sodium
            hydroxide in reagent water and dilute to 1 L.

7.1.6.3   Sulfuric acid, 50% (v/v) – Carefully add a portion of
            concentrated (95-98%) sulfuric acid to an equal portion of
            reagent water.

7.1.6.4   Magnesium chloride solution (2.5M) – Weigh 510 g of $MgCl_2 \bullet 6H_2O$
            into a 1000 milliliter (mL) flask, dissolve, and dilute to 1 L
            with reagent water.

7.1.6.5   Sulfamic acid (Powdered).

7.1.7   Micro-Distillation Reagents

7.1.7.1   Sodium hydroxide solution, 0.25N – Dissolve 10.0 g sodium
            hydroxide in reagent water and dilute to 1 L.

7.1.7.2   Sodium hydroxide solution, 1.25N - Dissolve 50.0 g sodium
            hydroxide in reagent water and dilute to 1 L.

7.1.7.3   Sulfuric Acid/Magnesium Chloride solution (7.11 M sulfuric acid/0.79 M magnesium chloride) - In a fume hood, weigh 32.2 g MgCl$_2$•6H$_2$0 into a tared 500 mL beaker and add 110.8 g reagent water. Add 139 g concentrated (95-98%) sulfuric acid in 40 g portions with stirring. Allow the solution to cool.

7.1.7.4   Sulfamic acid (Powdered).

7.1.8   Analytical Reagents

7.1.8.1   Chloramine-T solution (0.014M) - Dissolve 0.40 g of chloramine-T in reagent water and dilute to 100 mL. Prepare fresh daily.

7.1.8.2   Sodium dihydrogen Phosphate Buffer – Dissolve 138g of NaH$_2$PO$_4$•H$_2$O in 1L reagent water.

7.1.8.3   Pyridine-barbituric acid solution - Transfer 15 g of barbituric acid into a 1 L volumetric flask. Add about 100 mL of reagent water and swirl the flask. Add 75 mL of pyridine and mix. Add 15 mL of concentrated hydrochloric acid and mix.

Dilute to about 900 mL with reagent water and mix until the barbituric acid is dissolved. Dilute to 1 L with reagent water. Store at 4°C (±2°C).

7.2   Standards

7.2.1   Introduction

The Contractor must provide all standards, except as noted, to be used with this contract. These standards may be used only after they have been certified according to the procedure in Exhibit D – Introduction to Inorganic Analytical Methods, Section 11.0. The Contractor must be able to verify that the standards are certified. Manufacturer's certificates of analysis must be retained by the Contractor and presented upon request.

Samples, sample distillates, and standards must be stored separately.

7.2.2   Stock Standard Solutions

7.2.2.1   Stock cyanide solution, 1000 mg/L CN - Dissolve 2.51 g of potassium cyanide and 2.0 g potassium hydroxide in reagent water and dilute 1 L. Standardize with 0.0192N silver nitrate. Standardization is not necessary if this standard is purchased as a certified solution.

7.2.2.2   Intermediate cyanide standard solution, 10 mg/L CN - Dilute 1.0 mL of stock cyanide solution plus 20 mL of 1.25N sodium hydroxide solution to 100 mL with reagent water. Prepare this solution at time of analysis.

7.2.3   Secondary Dilution Standards

Prepare secondary dilution standard solutions by diluting the appropriate volumes of the intermediate cyanide standard solution with 0.25N sodium hydroxide. The final concentration of sodium hydroxide in all standards should be 0.25N.

7.2.4   Initial Calibration Verification Solution

7.2.4.1   The Initial Calibration Verification (ICV) solution shall be obtained from the EPA.

7.2.4.1.1   If the solution is not available from the EPA, then the ICV solution shall be prepared by the laboratory using a certified solution from an independent source. An independent source is defined as a standard from a different source than those used in the standards for the instrument calibration. Analyses shall be conducted at a concentration other than that used for instrument calibration, but near the middle of the calibration range.

7.2.4.1.2   The ICV standard shall be distilled in the same matrix as the calibration standards and in accordance with the instructions provided by the supplier.

7.3   Blanks

Two types of blanks are required for this method. A Calibration Blank is used to establish the analytical calibration curve and the Preparation Blank (see Section 12.1) is used to assess possible contamination from the sample preparation procedure and to assess spectral background.

7.3.1   Calibration Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Calibration Blank must be carried through the complete procedure and contain the same reagent concentration in the final solution as the sample solution used for analysis. The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) are identical in composition to the Calibration Blank and are used to monitor for analyte carryover.

7.3.2   Preparation Blank - Must contain all the reagents in the same volumes as used in preparing the samples. The Preparation Blank must be carried through the complete procedure and contain the same reagent concentration in the final solution as the sample solution used for analysis. Soil/sediment blanks shall use 1.00 mL (±0.01 mL) of reagent water.

8.0   SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1   Sample Collection and Preservation

All aqueous/water and soil/sediment samples must be collected in polyethylene or glass containers. The aqueous/water samples must be preserved with sodium hydroxide to pH greater than or equal to 10. All samples must be maintained at $\leq 6^{\circ}C$ but not frozen from the time of collection until receipt at the laboratory.

8.2   Sample Storage

All aqueous/water and soil/sediment samples must be protected from light and refrigerated at $\leq 6^{\circ}C$ but not frozen from the time of receipt until distillation.

8.2.1   Unused Sample Storage

The Contractor shall retain the unused portion of the aqueous/water and soil/sediment samples for a period of 60 days after delivery of a complete, reconciled data package to the EPA. The unused portion must be protected from light and refrigerated at $\leq 6^{\circ}C$ but not frozen.

8.2.2   Distillate Sample Storage

Distillates shall not be retained. Any reanalyses of the sample shall be performed using a freshly distilled aliquot of the sample.

8.2.3   Container Storage

The Contractor shall retain the empty sample containers for 60 days after the delivery of a complete, reconciled data package to the EPA. The sample container may be photographed in lieu of retention.

8.3   Contract Required Holding Time

The sample holding time for cyanide is 12 days from Validated Time of Sample Receipt (VTSR) to analysis. The holding time for the analysis of SPLP leachates is 12 days from the date of extraction.

9.0   CALIBRATION AND STANDARDIZATION

9.1   Instrument Operating Parameters

Because of the difference between various manufacturers and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. The Method Detection Limit (MDL) and precision must be investigated and established for cyanide on that particular instrument. All measurements must be within the operational range of the instrument. It is the responsibility of the analyst to verify the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain Quality Control (QC) data confirming instrument performance and analytical results.

9.2   Pre-Calibration Routine

Prior to calibration, the Contractor shall set up each instrument with the operating parameters established in Section 9.1 and conduct any necessary adjustments. The instrument must be allowed to become stable (usually 30 minutes) before calibration is performed. Establish a steady reagent baseline, adjusting as necessary.

9.3   Instrument Calibration Procedure

9.3.1   Summary of Instrument Calibration

Prior to sample analysis, the Contractor shall calibrate each instrument to determine sensitivity and linearity of the response.

9.3.2   Frequency of Instrument Calibration

Each instrument shall be calibrated daily or once every 24 hours, each time the instrument is set up, or after ICV, ICB, Continuing Calibration Verification (CCV), or CCB failure. The instrument calibration date and time shall be included in the raw data.

9.3.3   Procedure for Instrument Calibration

9.3.3.1   Each instrument shall be calibrated according to the manufacturer's recommended procedures.

9.3.3.2   At least six calibration standards shall be used. The calibration standards shall be prepared according to Sections 7.2 and 10.2. One of the standards shall be a blank standard and one shall be at or below the Contract Required Quantitation Limit (CRQL), but greater than the MDL. The rest of the standards shall be uniformly spread over the appropriate calibration range.

9.3.3.3   Calibration standards shall be distilled fresh with each calibration performed according to Section 10.2.

9.3.4   Calculations for Instrument Calibration

9.3.4.1   The calibration curve shall be calculated using linear regression by plotting the concentration of the standard [in micrograms/Liter (μg/L)] on the X-axis versus the instrument response (e.g., absorbance) on the Y-axis.

9.3.4.2   The Contractor may use standard linear regression, weighted linear regression (1/concentration, 1/square of concentration, variance, 1/variance, standard deviation, 1/standard deviation, or 1/square of standard deviation), or linear regression with zero force calibration models, as appropriate for the above calculation. No other types of equations (e.g., quadratic) are to be used.

9.3.4.3   The calibration curve shall be checked to establish the representativeness of the data that were used to produce it. This check involves re-fitting the data of the non-blank calibration standards back to the calibration curve, and computing the difference between the calculated concentration and the expected concentration for each of these standards. The difference is then divided by the expected concentration of the respective standard and multiplied by 100.

9.3.5   Technical Acceptance Criteria for Instrument Calibration

9.3.5.1   The correlation coefficient of the calibration curve shall be greater than or equal to 0.995.

9.3.5.2   The Percent Difference (%D) for each of the standards shall be within the control limits of ±30%.

9.3.5.3   If a standard is analyzed at a concentration that is below the CRQL and the %D criteria is not met, that standard can be excluded from the calibration curve as long as the lowest non-zero standard is still analyzed at or below the CRQL and all standards included in the calibration curve are continuous and consecutive.

9.3.6   Corrective Action for Instrument Calibration

9.3.6.1   Sample analysis shall not begin until the criteria described in Section 9.3.5 have been met.

9.3.6.2   Any changes or corrections to the analytical system shall be followed by recalibration.

9.4   Initial Calibration Verification

9.4.1   Summary of Initial Calibration Verification

Prior to sample analysis, the Contractor shall demonstrate the accuracy of the instrument calibration for each instrument through the analysis of an ICV standard.

9.4.2   Frequency of Initial Calibration Verification

The ICV shall be analyzed immediately after the instrument has been calibrated.

9.4.3   Procedure for Initial Calibration Verification

9.4.3.1   The ICV shall be analyzed at the wavelength used to report final results.

9.4.3.2   The ICV shall reflect the conditions of analysis of the associated analytical samples.

9.4.4   Calculations for Initial Calibration Verification

The Percent Recovery (%R) of the ICV shall be calculated using the following equation:

EQ. 1 ICV Percent Recovery

$$\%R = \frac{\text{Found(ICV)}}{\text{True(ICV)}} \times 100$$

WHERE,

Found (ICV) = The found concentration of cyanide in the ICV Solution

True (ICV)   = The expected concentration of cyanide in the ICV Solution

9.4.5   Technical Acceptance Criteria for Initial Calibration Verification

The ICV %R shall be within the control limits of 85-115%.

9.4.6   Corrective Action for Initial Calibration Verification

If the recovery is outside the control limits of 85% Recovery (low) or 115% Recovery (high), the analysis shall be terminated, the problem corrected, the instrument recalibrated, and the calibration verified.

9.5   Continuing Calibration Verification

9.5.1   Summary of Continuing Calibration Verification

Prior to sample analysis, periodically during analysis, and at the conclusion of analysis, the Contractor shall demonstrate the stability of the calibration through the analysis of a CCV standard. The Contractor shall not analyze a blank immediately prior to the analysis of the CCV.

9.5.2   Frequency of Continuing Calibration Verification

9.5.2.1   A CCV shall be analyzed at the beginning of the analytical sequence and after the last analytical sample. A CCV shall also be analyzed at a frequency not to exceed 1 hour during an analytical sequence. See the example analytical sequence in Section 10.5.6.

9.5.2.2   The analytical sequence can continue for 24 hours as long as samples are being continuously analyzed without the instrument being turned off and successive CCV standards meet the technical acceptance criteria in Section 9.5.5.

9.5.3   Procedure for Continuing Calibration Verification

9.5.3.1   The CCV standard shall be prepared using the same source and in the same acid matrix as the calibration standards at or near to the mid-level of the calibration curve. The CCV shall be prepared according to Section 10.0.

9.5.3.2   The same CCV standard shall be used throughout the analytical sequences for an SDG.

9.5.3.3   The CCV shall be analyzed at the wavelength used to report final results.

9.5.3.4   Each CCV analyzed shall reflect the conditions of analysis of all associated analytical samples (the preceding analytical samples up to the previous CCV as applicable and the analytical samples following this CCV up to the next CCV as applicable).

9.5.4   Calculations for Continuing Calibration Verification

The %R of the CCV shall be calculated using the following equation:

EQ. 2 CCV Percent Recovery

$$\%R = \frac{\text{Found (CCV)}}{\text{True(CCV)}} \times 100$$

WHERE,

Found (CCV) = The found concentration of cyanide in the
                CCV Solution
True (CCV)   = The expected concentration of cyanide in the
                CCV Solution

9.5.5   Technical Acceptance Criteria for Continuing Calibration
         Verification

9.5.5.1   The CCV %R shall be within the control limits of 85-115%.

9.5.5.2   All samples shall be analyzed within 1 hour of an acceptable
           opening and closing CCV.

9.5.6   Corrective Action for Continuing Calibration Verification

If the deviations of the CCV are greater than the specified control
limits of 85% Recovery (low) or 115% Recovery (high), the analysis
shall be terminated, the problem corrected, the instrument
recalibrated, the calibration verified, and reanalysis of all
analytical samples analyzed since the last compliant calibration
verification shall be performed.

9.6   Initial and Continuing Calibration Blank

9.6.1   Summary of Calibration Blank

Prior to sample analysis, periodically during analysis, and at the
conclusion of analysis, the Contractor shall demonstrate the
stability of the calibration baseline through the analysis of an ICB
and CCB.

9.6.2   Frequency of Calibration Blank

9.6.2.1   An ICB shall be analyzed immediately after the instrument has
           been calibrated and the calibration verified by the analysis of
           the ICV.

9.6.2.2   Each CCB shall be analyzed immediately after the analysis of a
           CCV.

9.6.3   Procedure for Calibration Blank

9.6.3.1   The ICB and CCB samples shall be analyzed at the wavelength used
           for reporting final results.

9.6.3.2   The ICB analyzed shall reflect the conditions of analysis of the
           associated analytical samples.

9.6.3.3   Each CCB analyzed shall reflect the conditions of analysis of the
           associated analytical samples (the preceding analytical samples
           up to the previous CCB as applicable and the analytical samples
           following this CCB up to the next CCB as applicable).

9.6.4   Calculations for Calibration Blank

The results for the ICB and CCB samples shall be calculated using
Equation 3 in Section 11.0.

9.6.5   Technical Acceptance Criteria for Calibration Blank

The absolute value of each calibration blank result shall be less than or equal to the CRQL for aqueous/water samples.

9.6.6   Corrective Action for Calibration Blank

If the absolute value of the calibration blank exceeds the CRQL for aqueous/water samples, the analysis shall be terminated, the problem corrected, the instrument recalibrated, the calibration verified, and reanalysis of all affected analytical samples analyzed since the last compliant calibration blank performed.

10.0   PROCEDURE

10.1   Pre-Distillation Sample Preparation

10.1.1   The Contractor shall measure the sample pH at the time of sample receipt to verify that the sample was properly preserved (see Exhibit D – General Inorganic Analysis, Section 10.1.2.1).

10.1.2   Before preparation is initiated for an aqueous/water sample, the Contractor shall test for the presence of sulfides and oxidizing agents (e.g., residual chlorine). The Contractor shall document the presence of sulfides or oxidizing agents in the SDG Narrative. The Contractor shall document the results (positive or negative) of the tests for sulfides and oxidizing agents in the distillation log.

10.1.2.1   The test for sulfides shall be performed by placing a drop of the sample on a strip of lead acetate paper. If the test strip turns black, the Contractor shall contact the Sample Management Office (SMO) for further instructions from the EPA Region before proceeding with sample preparation and analysis.

10.1.2.2   The test for oxidizing agents shall be performed by placing a drop of the sample on a strip of potassium iodide – starch test paper (KI - starch paper). If the test strip turns blue, the Contractor shall contact SMO for further instructions from the EPA Region before proceeding with sample preparation and analysis.

10.2   Standards Preparation

All standards for the midi-distillation and micro-distillation semi-automated spectrophotometric analysis shall be distilled in the same manner as the samples.

10.2.1   Prepare at least five standards and a calibration blank according to Section 9.3.

NOTE: The concentration of one of the calibration standards shall be at or below the CRQL, but greater than the MDL.

10.2.2   For midi-distillation, the standards shall be prepared by pipetting suitable volumes of the secondary dilution standard solution (see Section 7.2.3) into volumetric flasks and diluting to volume with 0.25N sodium hydroxide. Add 50 mL of each standard to a midi-distillation tube and then prepare and distill these standards and the calibration blank in the same manner as the samples.

10.2.3   For micro-distillation, the standards shall be prepared by pipetting suitable volumes of the secondary dilution standard solution (see Section 7.2.3) into volumetric flasks and diluting to volume with 0.25N sodium hydroxide. Add 6 mL of each standard to a sample tube and then prepare and distill these standards and the calibration blank in the same manner as samples.

10.3 Aqueous/Water Sample Preparation

10.3.1   Preparation Method by Midi-Distillation [based on EPA Method 335.4, Revision 1.0 (August 1993)]

10.3.1.1   Pipet 50 mL (±1 mL) of sample into the distillation flask along with 2 or 3 boiling chips (as necessary). The sample shall not be diluted prior to distillation.

10.3.1.2   Add 50 mL (±1 mL) of 0.25N sodium hydroxide to the gas absorbing tube.

10.3.1.3   Connect the boiling flask, condenser, and absorber in the train. The excess cyanide trap contains 0.5N sodium hydroxide.

10.3.1.4   Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of between 2 to 3 bubbles per second from the impingers in each reaction vessel.

10.3.1.5   Test sample for nitrate and/or nitrite using an appropriate test strips or kits. Record method, manufacturer information, and results on the Distillation Log and in the SDG Narrative. If the samples contain nitrate and/or nitrite, add 0.2 g of sulfamic acid through the air inlet tube. The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid prior to the distillation. Mix for 3 minutes prior to adding the sulfuric acid.

10.3.1.6   After 5 minutes of vacuum flow, inject 5 mL of 50% (v/v) sulfuric acid through the top air inlet tube of the distillation head into the reaction vessel. Allow the airflow to mix the reaction vessel contents for 5 minutes.

NOTE: The acid volume must be sufficient to bring the sample/solution pH to below 2.0.

10.3.1.7   Add 2 mL of the 2.5M magnesium chloride solution through the top air inlet tube of the distillation head into the reaction vessel. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of the 2.5M magnesium chloride solution or a few drops of a commercially available anti-foam agent. The Contractor shall document the addition of magnesium chloride solution or anti-foam agent in the SDG Narrative.

10.3.1.8   Turn on the heating block and set for 125°C (±3°C). Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

10.3.1.9   After 1 1/2 hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

10.3.1.10 After cooling, close off the vacuum at the gang valve and remove the absorber. Seal the distillate and store at 4°C until analyzed.

10.3.2   Preparation Method by Micro-Distillation (based on Lachat QuikChem Method 10.204-00-1-X)

10.3.2.1   Preheat the heater block to 120°C (±3°C).

10.3.2.2   Add 6 mL (±0.1 mL) of sample to the sample tube. The sample shall not be diluted prior to distillation. If the Contractor is not using the prefilled collector tubes, add 2 mL (±0.1 mL) of the 0.25N absorbing solution to each collector tube. Add 0.75 mL of the (7.11M/0.79M) sulfuric acid/magnesium chloride solution to each sample tube and immediately cap with a collector tube and press to seal.

10.3.2.3   Place the assembled tubes into the heater block and heat for 30 minutes. After 30 minutes, remove each tube from the block and immediately pull off the sample tube.

10.3.2.4   Invert each collector tube and allow to cool. Mix the distillate and detach the upper portion. Dilute the distillate to 6 mL (±0.1 mL) with absorbing solution and mix. Seal the distillate and store at 4°C until analyzed.

10.4 Soil/Sediment Sample Preparation

10.4.1   Preparation Method by Midi-Distillation [based on EPA Method 335.4, Revision 1.0 (August 1993)]

10.4.1.1   Mix the sample thoroughly to achieve homogeneity. Weigh to the nearest 0.01 g and transfer 1.00-1.50 g of sample (wet weight) into the reaction vessel and add 50 mL of reagent water. Add 2 or 3 boiling chips (as necessary).

10.4.1.2   Add 50 mL (±1 mL) of 0.25N sodium hydroxide to the gas absorbing impinger.

10.4.1.3   Connect the reaction vessel, condenser, and absorber in the train. The excess cyanide trap contains 0.5N sodium hydroxide.

10.4.1.4   Turn on the vacuum and adjust the gang (Whitney) valves to give a flow of between 2 to 3 bubbles per second from the impingers in each reaction vessel.

10.4.1.5   After 5 minutes of vacuum flow, inject 5 mL of 50% (v/v) sulfuric acid through the top air inlet tube of the distillation head into the reaction vessel. Allow the airflow to mix the reaction vessel contents for 5 minutes.

NOTE: The acid volume must be sufficient to bring the sample/solution pH to below 2.0.

10.4.1.6   Add 2 mL of the 2.5M magnesium chloride solution through the top air inlet tube of the distillation head into the reaction vessel. Excessive foaming from samples containing surfactants may be quelled by the addition of either another 2 mL of the 2.5M magnesium chloride solution or a few drops of a commercially available anti-foam agent. The Contractor shall document the addition of the magnesium chloride solution or anti-foam agent in the SDG Narrative.

10.4.1.7   Turn on the heating block and set for 125°C (±3°C). Heat the solution to boiling, taking care to prevent solution backup by periodic adjustment of the vacuum flow.

10.4.1.8   After 1 1/2 hours of refluxing, turn off the heat and continue the vacuum for an additional 15 minutes. The flasks should be cool at this time.

10.4.1.9   After cooling, close off the vacuum at the gang valve and remove the absorber. Seal the distillate and store at 4°C until analyzed.

10.4.2   Preparation Method by Micro-Distillation (based on Lachat QuikChem Method 10-204-00-1-X)

10.4.2.1   Preheat the heater block to 120°C (±3°C).

10.4.2.2   Add 0.50-1.00 g (±0.01 g) of sample (wet weight) and 5 mL of reagent water to the sample tube. If the Contractor is not using the prefilled collector tubes, add 2 mL (±0.1 mL) of the 0.25N absorbing solution to the collector tube. Add 0.75 mL of the (7.11M/0.79M) sulfuric acid/magnesium chloride solution to each sample tube and immediately cap with collector tube and press to seal.

10.4.2.3   Place the assembled tubes into the heater block and heat for 30 minutes. After 30 minutes, remove each tube from the block and immediately pull off the sample tube.

10.4.2.4   Invert each collector tube and allow to cool. Mix the distillate and detach the upper portion. Dilute the distillate to 6 mL (±0.1 mL) with absorbing solution and mix. Seal the distillate and store at 4°C until analyzed.

10.5 Sample Analysis

10.5.1   Set up the manifold. Pump the reagents through the system until a steady baseline is obtained.

10.5.2   Place the distilled calibration standards, blanks, and control standards in the sampler tray, followed by the distilled samples, duplicates, standards, spikes, and blanks. See example sequence provided in Section 10.5.6.

10.5.3   Allow all standards and samples to come to ambient room temperature prior to analysis.

10.5.4   When a steady reagent baseline is obtained and before starting the sampler, adjust the baseline using the appropriate knob on the spectrophotometer. Aspirate the distilled blank calibration standard and adjust the spectrophotometer until the desired signal is obtained. Establish the baseline and proceed to analyze the remainder of the distilled standards and distilled samples.

10.5.5   Sample distillates having concentrations higher than the established calibration range as determined by the expected concentration of the highest calibration standard shall be diluted into range with the absorbing solution and reanalyzed.

10.5.6   Example Analytical Sequence for Cyanide Including the Instrument Calibration:

S##
S##
S##
S##
S##
S##
ICV
ICB
CCV###
CCB###
samples
CCV###
CCB###
samples
CCV###
CCB###, etc.

11.0   DATA ANALYSIS AND CALCULATIONS

Calculate the Cyanide concentration using the following equations.

11.1   Aqueous/Water and SPLP Leachate Sample Calculation

EQ. 3 Aqueous/Water and SPLP Leachate Sample Concentration

$$\text{CN Concentration (µg/L)} = C \times \frac{V_f}{V} \times DF$$

WHERE,
C   =   Instrument response in µg/L CN from the calibration curve
V_f =   Final prepared (absorbing solution) volume (mL)
V   =   Initial aliquot amount (mL)
DF   =   Dilution Factor

11.2   Soil/Sediment Sample Calculation

The concentrations in the distillates are to be reported on the basis of the dry weight of the sample, in units of milligram/kilogram (mg/kg):

EQ. 4 Soil/Sediment Sample Concentration

$$\text{CN Concentration (mg/kg dry weight)} = C \times \frac{V_f}{W \times S} \times (1/1000) \times DF$$

WHERE,
C   =   Instrument response in µg/L CN from the calibration curve
V_f =   Final prepared (absorbing solution) volume (mL)
W   =   Initial aliquot amount (g)
S   =   % Solids/100 (see Exhibit D – General Inorganic Analysis, Section 10.1.1)
DF   =   Dilution Factor

11.3   Adjusted Contract Required Quantitation Limit Calculation

11.3.1   Calculate the adjusted CRQL for aqueous/water or SPLP leachate samples by multiplying the CRQL (µg/L) by the sample dilution factor and the V_f/V terms as noted in Equation 3.

11.3.2   Calculate the adjusted CRQL for soil/sediment using the following equation:

EQ. 5 Adjusted Soil/Sediment CRQL

$$\text{Adjusted CRQL (mg/kg)} = C \times \frac{W_M}{W \times S} \times DF$$

WHERE,
C   =   CRQL (mg/kg)
W_M =   Minimum method required aliquot amount (1.00 g for midi or 0.50 g for micro)
W   =   Initial aliquot amount (g)
S   =   % Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.1)
DF   =   Dilution Factor

12.0 QUALITY CONTROL

12.1 Preparation Blank Sample

12.1.1   Summary of Preparation Blank Sample

The Preparation Blank is used to monitor for possible contamination throughout the entire sample preparation and analysis process.

12.1.2   Frequency of Preparation Blank Sample

At least one Preparation Blank shall be prepared with every SDG, or with each preparation batch, whichever is more frequent.

12.1.3   Procedure for Preparation Blank Sample

The Preparation Blank shall be carried through the complete sample preparation procedure for the matrix and contain the same reagent concentration in the final distillate as the samples.

12.1.4   Calculations for Preparation Blank Sample

Calculate the results for aqueous Preparation Blanks by using Equation 3. Calculate the results for soil/sediment Preparation Blanks by using Equation 4.

12.1.5   Technical Acceptance Criteria for Preparation Blank Sample

12.1.5.1   The absolute value of the Preparation Blank result shall be less than or equal to the CRQL.

12.1.5.2   The cyanide concentration in the Preparation Blank may be greater than the CRQL, if the concentration of cyanide in the associated samples is greater than or equal to 10 times the blank concentration.

12.1.5.3   The cyanide concentration in the Preparation Blank may be less than the negative CRQL if the concentration in the associated samples is greater than or equal to 10 times the CRQL.

12.1.6   Corrective Action for Preparation Blank Sample

12.1.6.1   If the cyanide concentration in the Preparation Blank is greater than the CRQL, and the concentration of cyanide in any of the associated samples is less than 10 times the blank concentration, then all samples with less than 10 times the blank concentration shall be reprepared and reanalyzed with appropriate new QC.

12.1.6.2   If the cyanide concentration in the Preparation Blank is less
            than the negative CRQL and the concentration in the associated
            samples is less than 10 times the CRQL, then all samples with
            less than 10 times the CRQL concentration shall be reprepared and
            reanalyzed with appropriate new QC.

12.2 Matrix Spike and Post-Distillation Spike Samples

12.2.1   Summary of Matrix Spike and Post-Distillation Spike Samples

          The Matrix Spike sample analysis is designed to provide information
          about the effect of the sample matrix on the distillation and/or
          measurement methodology.

12.2.2   Frequency of Matrix Spike and Post-Distillation Spike Samples

12.2.2.1   At least one Matrix Spike sample analysis shall be performed on
            each group of samples of a similar matrix type (i.e.,
            aqueous/water, soil/sediment) or for each SDG, whichever is more
            frequent.[1]

12.2.2.2   If a Matrix Spike sample does not meet the technical acceptance
            criteria listed in Section 12.2.5, a Post-Distillation Spike
            Sample shall be performed for those analytes that do not meet the
            specified criteria.

12.2.3   Procedure for Matrix Spike and Post-Distillation Spike Samples

12.2.3.1   For a Matrix Spike sample, the spike is added before the
            distillation (i.e., prior to the addition of other reagents).

12.2.3.2   The analyte spike shall be added to achieve a concentration of
            100 µg/L in the final sample solution prepared for analysis
            (i.e., post-distillation). For example, the midi-distillation
            procedure would require the addition of 5 µg of cyanide to the
            sample prior to distillation (based on the final distillation
            volume of 50 mL). For a typical 50 mL aqueous/water sample, this
            would be equivalent to a concentration of 100 µg/L in the
            original sample. For a typical 1.00 g soil/sediment sample, this
            would be equivalent to a concentration of 5 mg/kg in the original
            dry sample. Adjustments shall be made to maintain these spiking
            levels when the weight of the sample taken deviates by more than
            10% of these values.

12.2.3.3   For a Post-Distillation Spike sample, the sample that was
            initially used for the Matrix Spike analysis shall be used for
            the Post-Distillation Spike analysis. Spike the unspiked aliquot
            of the original distillate at two times the indigenous level of
            two times the CRQL, whichever is greater.

12.2.3.4   Samples identified as field blanks and Performance Evaluation
            (PE) samples shall not be used for Matrix Spike sample analysis.
            The EPA may require that a specific sample be used for the Matrix
            Spike sample analysis.

[1] The EPA may require additional spike sample analyses, upon request from the
EPA Regional Contract Laboratory Program Contracting Officer's Representative
(EPA Regional CLP COR).

12.2.4   Calculations for Matrix Spike and Post-Distillation Spike Samples

12.2.4.1   If the Matrix Spike analysis is performed on the same sample that is chosen for the Duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample" (see Section 12.3). The average of the duplicate results cannot be used for the purpose of determining %R.

12.2.4.2   Calculate the Matrix Spike and Post-Distillation Spike %R using the following equation:

EQ. 6 Matrix Spike and Post-Distillation Spike Percent Recovery

$$\%R = \frac{SSR - SR}{SA} \times 100$$

WHERE,
SSR = Spiked sample result (original) (µg/L or mg/kg) from EQ. 3 or EQ. 4
SR   = Sample result (original) (µg/L or mg/kg) from EQ. 3 or EQ. 4. When the sample concentration is less than the MDL, use SR=0.
SA   = Spike Added Theoretical Result (µg/L or mg/kg). This is calculated by using the spike concentration specified in Section 12.2.3.2 and applying all corrections used in calculating the sample concentration.

NOTE: The units used for reporting SSRs will be identical to those used for reporting SRs.

12.2.5   Technical Acceptance Criteria for Matrix Spike and Post-Distillation Spike Samples

The Matrix Spike and Post-Distillation Spike %R shall be within the control limits of 75-125%.

12.2.6   Corrective Action for Matrix Spike Sample

12.2.6.1   If the Matrix Spike recovery is not within the control limits of 75-125%, the data for all samples received and associated with that spike sample shall be flagged with "*". An exception to this rule is granted when the sample concentration exceeds the SA concentration by a factor of four or more. In such an event, the data shall be reported unflagged even if the %R does not meet the 75-125% recovery criteria.

12.2.6.2   When the Matrix Spike recovery is outside the control limits and the sample result does not exceed four times the spike added, a Post-Distillation spike shall be performed following procedures in Section 12.2.3.

12.2.6.3   If there is more than one Matrix Spike per matrix, per SDG, if one Matrix Spike sample recovery is not within contract criteria, then flag all the samples of the same matrix and method in the SDG.

12.3 Duplicate Sample

12.3.1   Summary of Duplicate Sample

Duplicates are analyzed to help determine sample homogeneity and laboratory precision.

12.3.2   Frequency of Duplicate Sample

12.3.2.1   One duplicate sample shall be analyzed from each group of samples of a similar matrix type (i.e., aqueous/water, soil/sediment) or for each SDG, whichever is more frequent.[2]

12.3.2.2   Duplicate sample analyses cannot be averaged for reporting.

12.3.3   Procedure for Duplicate Sample

12.3.3.1   Samples identified as field blanks and PE samples shall not be used for duplicate sample analysis. The EPA may require that a specific sample be used for duplicate sample analysis.

12.3.3.2   Prepare a second aliquot of the original sample. The duplicate sample shall be carried through the complete sample preparation procedure.

12.3.4   Calculations for Duplicate Sample

12.3.4.1   The Relative Percent Difference (RPD) for each analyte shall be calculated using the following equation:

EQ. 7 Duplicate Sample Relative Percent Difference

$$RPD = \frac{\left| S - D \right|}{(S+D)/2} \times 100$$

WHERE,
S   =   Sample Result (original) (µg/L or mg/kg) from EQ. 3 or EQ. 4
D   =   Duplicate Sample Result (µg/L or mg/kg) from EQ. 3 or EQ. 4

12.3.5   Technical Acceptance Criteria for Duplicate Sample

12.3.5.1   The RPD shall be within the control limits of ±20 if the original and duplicate sample values are greater than or equal to five times the CRQL (see Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits).

12.3.5.2   The control limit shall be equal to the CRQL if either the sample or duplicate value are less than five times the CRQL, or if one result is above five times the CRQL level and the other is below.

12.3.5.3   If both sample and duplicate values are less than the CRQL, the RPD is not calculated.

12.3.6   Corrective Action for Duplicate Sample

12.3.6.1   If the duplicate sample results are outside the control limits, flag all the data for samples received associated with that duplicate sample with an "*".

12.3.6.2   If there is more than one duplicate sample per matrix per SDG, and if one duplicate result is not within contract criteria, flag all samples of the same matrix in the SDG.

[2] The EPA may require additional duplicate sample analyses, upon request from the EPA Regional CLP COR.

12.4 Method Detection Limit Determination

12.4.1 Before any field samples are analyzed, the MDLs shall be determined for each distillation procedure and instrument used prior to the start of contract analyses and annually thereafter. An MDL study shall be performed after major instrument maintenance, or changes in instrumentation or instrumental conditions, to verify the current sensitivity of the analysis.

12.4.1.1 To determine the MDLs, the Contractor shall perform MDL studies following the procedures in 40 Code of Federal Regulations (CFR), Part 136.

12.4.1.2 The Contractor shall prepare the MDL samples by each distillation procedure used and shall analyze these samples on each instrument used.

12.4.1.3 The determined concentration of the MDL shall be less than half the concentration of the CRQL listed in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

12.5 Summary of Quality Control Operations

The QC operations performed for cyanide analysis are summarized in Table 1 – QC Operations for Cyanide Analysis.

13.0 METHOD PERFORMANCE

Not applicable.

14.0 POLLUTION PREVENTION

See Section 13.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

15.0 WASTE MANAGEMENT

See Section 14.0 in Exhibit D - Introduction to Inorganic Analytical Methods.

16.0 REFERENCES

16.1 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 335.4, Revision 1.0, August 1993.

16.2 American Water Works Association/American Public Health Association/Water Environment Federation, Standard Methods for the Examination of Water and Wastewater, Method 4500-CN E.

16.3 U.S. Government Printing Office, 40 Code of Federal Regulations, Part 136, Section 1, Appendix B.

16.4 Lachat QuikChem Method 10-204-00-1-X.

17.0   TABLES/DIAGRAMS/FLOWCHARTS

TABLE 1. QC OPERATIONS FOR CYANIDE ANALYSIS

QC Operation	Frequency
Instrument Calibration	Daily or each time instrument is turned on or set up, after ICV or CCV failure, and after major instrument adjustment.
Initial Calibration Verification	Following each instrument calibration.
Continuing Calibration Verification	At a frequency of every hour of an analytical sequence, and at the beginning and end of each analytical sequence.
Initial Calibration Blank	Following each instrument calibration, immediately after the ICV.
Continuing Calibration Blank	Every hour and at the beginning and end of each analytical sequence. Performed immediately after the CCV.
Preparation Blank	For each SDG or each sample preparation and analysis procedure per batch of prepared samples, whichever is more frequent.
Matrix Spike Sample	For each matrix type or for each SDG, whichever is more frequent.
Post-Distillation Spike	Each time Spike Sample Recovery is outside QC limits.
Duplicate Sample Analysis	For each matrix type or for each SDG, whichever is more frequent.
Method Detection Limit Determination	Prior to start of contract analyses, annually thereafter, and after major instrument adjustment.

EXHIBIT E

QUALITY SYSTEMS

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Exhibit E – Quality Systems

Table of Contents

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1.0   QUALITY SYSTEM

1.1   Overview

Since the purpose of this analytical service is to provide analytical data for the use by the U.S. Environmental Protection Agency (EPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act (SARA), the Contractor is responsible for developing and implementing a Quality System to enforce the requirements of the EPA CIO 2105.0 "*Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*". This will require the implementation of a quality system that meets the EPA's goal of providing data of documented quality.

1.1.1   The quality system provides the framework for planning, implementing, assessing, and improving work performed by the Contractor for performing quality assurance (QA) and quality control (QC) activities. Effective implementation of the quality system leads to several benefits including:

- Scientific Data Integrity – The Contractor will produce and submit data of known and documented quality;

- Effective Management of Internal and External Activities – The quality system requires documentation of activities and oversight for evaluation purposes which will reduce the potential for waste and abuse; and

- Continual Improvement – The continual improvement component of the quality system leads to the development of a better more responsive quality system and technical system which should result in better products and services.

1.1.2   Overall, successful implementation of the quality system will reduce the Agency's vulnerabilities in decision making and increase the EPA's credibility by providing the ability to make reliable, timely, cost effective, and defensible decisions. The consequences of not having a successfully implemented quality system include the potential to waste time, money, and resources, which increase uncertainty in the EPA's decision.

1.1.3   Under this program, the EPA requires two forms of documentation for the quality system:

- A Quality Management Plan (QMP) which documents the organization quality system; and

- A Quality Assurance Project Plan (QAPP) which documents the application of quality related activities to an activity-specific effort.

    NOTE: The Contractor may combine these two documents into a single document that describes the organization's quality system and the application of this system to the work performed under this program.

2.0   QUALITY MANAGEMENT PLAN

During the contract solicitation process, the Contractor is required to submit the QMP or equivalent to the EPA Contracting Officer (CO). The QMP documents how an organization structures its quality system and describes its quality policies and procedures; criteria for and areas of application; and roles, responsibilities, and authorities. It also describes an organization's policies and procedures for implementing and assessing the effectiveness of the quality system. The Contractor shall follow the EPA Requirements for Quality Management Plans (QA/R-2) EPA/240/B-01/002 (or subsequent version) for guidance.

2.1   The QMP should describe the Quality System that is designed to support the objectives of the organization in providing the analytical services required in this document.

2.2   The QMP must be sufficiently inclusive, explicit, and readable to enable both management and staff to understand the priority which management places on QA and QC activities, established quality policies and procedures, and their respective quality related roles and responsibilities.

2.3   The QMP should document management practices, including QA and QC activities, used to ensure that the results of technical work are of the type and quality needed for their intended use.

2.4   The QMP should document the following: the mission and quality policy of the organization; the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities; the means by which effective communications with personnel actually performing the work are assured; the processes used to plan, implement, and assess the work performed; the process by which measures of effectiveness for QA and QC activities will be established and how frequently effectiveness will be measured; and the continual improvement based on lessons learned from previous experience.

2.5   The elements to be addressed in a QMP include: management and organization; quality system description; personnel qualifications and training; procurement of items and services; documentation and records; computer hardware and software; planning; implementation of work processes; assessment and response; and quality improvement.

NOTE: It is not necessary for the Contractor to present the information in the same order as outlined above as long as each item is adequately addressed in the plan.

3.0   QUALITY ASSURANCE PROJECT PLAN

3.1   Introduction

The EPA requires that all environmental data used in decision making be supported by an approved QAPP. The QAPP integrates all technical and quality aspects of a project including planning, implementation, and assessment. The purpose of the QAPP is to document how QA and QC are applied to an environmental data operation to assure that the results obtained are of the type and quality needed and expected for this program. The Contractor shall follow the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5 (EPA/240/B-01/003) (or subsequent version) for guidance.

3.1.1   The Contractor shall prepare a written QAPP which describes the procedures that are implemented to:

- Maintain data integrity, validity, and usability;

- Ensure that analytical measurement systems are maintained in an acceptable state of stability and reproducibility;

- Detect problems through data assessment and establish corrective action procedures which keep the analytical process reliable; and

- Document all aspects of the measurement process to provide data which are technically sound and legally defensible.

3.1.2   The QAPP must present, in specific terms, the policies, organization, objectives, functional guidelines, and specific QA and QC activities designed to achieve the data quality requirements in this contract. Where applicable, Standard Operating Procedures (SOPs) pertaining to each element shall be included or referenced as part of the QAPP.

3.1.3   The QAPP shall be available during on-site laboratory evaluations.

3.1.4   The QAPP shall be submitted within 7 days of written request by the EPA Regional Contract Laboratory Program Contracting Officer's Representative (EPA Regional CLP COR) or the Analytical Services Branch CLP COR (ASB CLP COR).

3.2   Required Elements of a Quality Assurance Project Plan

The QAPP shall be paginated consecutively in ascending order. The required elements of a laboratory's QAPP are outlined in this section. This outline should be used as a framework for developing the QAPP.

A.   Organization and Personnel

1.   QA Policy and Objectives (the mission and quality policy of the organization)

2.   QA Management (the specific roles, authorities, and responsibilities of management and staff with respect to QA and QC activities)

a.   Organization

b.   Assignment of QA/QC Responsibilities

c.   Reporting Relationships (the means by which effective communication with personnel actually performing the work are ensured)

d.   QA Document Control Procedures

        e.   QA Program Assessment Procedures (the process used to plan, implement, and assess the work performed)

    3.   Key Personnel (laboratory personnel involved in QA and QC activities)

        a.   Resumes

        b.   Education and Experience Relevant to this Contract

        c.   Training Records and Progress

B.   Facilities and Equipment

    1.   Instrumentation and Backup Alternatives

    2.   Maintenance Activities and Schedules

C.   Document Control

    1.   Laboratory Notebook Policy

    2.   Sample Tracking/Custody Procedures

    3.   Logbook Maintenance and Archiving Procedures

    4.   Complete Sample Delivery Group (SDG) File (CSF) Organization, Preparation, and Review Procedures

    5.   Procedures for Preparation, Approval, Review, Revision, and Distribution of SOPs

    6.   Process for Revision of Technical or Documentation Procedures

D.   Analytical Methodology

    1.   Calibration Procedures and Frequency

    2.   Sample Preparation/Extraction Procedures

    3.   Sample Analysis Procedures

    4.   Standards Preparation Procedures

    5.   Decision Processes, Procedures, and Responsibility for Initiation of Corrective Action

E.   Data Generation

    1.   Data Collection Procedures

    2.   Data Reduction Procedures

    3.   Data Validation Procedures

    4.   Data Reporting and Authorization Procedures

F.   QA (the process which measures the effectiveness of QA will be established and how frequently effectiveness will be measured)

    1.   Data QA

    2.   Systems/Internal Audits

    3.   Performance/External Audits

    4.   Corrective Action Procedures (the continual improvement based on lessons learned from previous experience)

    5.   QA Reporting Procedures

    6.   Responsibility Designation

      G.   QC

          1.   Solvent, Reagent, and Adsorbent Check Analysis

          2.   Reference Material Analysis

          3.   Internal QC Checks

          4.   Corrective Action and Determination of QC Limit Procedures

          5.   Responsibility Designation

3.3 Submission of the Quality Assurance Project Plan

3.3.1 Initial Submission

The Contractor is required to submit their QAPP to the EPA CO within the number of days provided in the associated laboratory contract document. The Contractor shall maintain a QAPP (fully compliant with the requirements of this contract) on file at their facility for the term of the contract.

3.3.2 Revision Submissions

The revised QAPP will become the official QAPP under the contract and may be used during legal proceedings.

3.3.2.1 During the term of the contract, the Contractor shall amend the QAPP when the following circumstances occur:

- The EPA modifies technical requirements of the Statement of Work (SOW) or the contract;

- The EPA notifies the Contractor of deficiencies in the QAPP document;

- The EPA notifies the Contractor of deficiencies resulting from the EPA's review of the Contractor's performance;

- The Contractor identifies changes in organization, personnel, facility, equipment, policy, or procedures; or

- The Contractor identifies deficiencies resulting from the internal review of their organization, personnel, facility, equipment, policy, procedure, or QAPP document.

3.3.2.2 The Contractor shall amend and submit the QAPP to the recipient(s) identified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule, within 14 days of when the circumstances listed above result in a discrepancy between what was previously described in the QAPP, and what is presently occurring at the Contractor's facility.

3.3.2.2.1 All changes in the QAPP shall be clearly marked (e.g., a bar in the margin indicating where the change is found in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font) and the amended section pages shall have the date on which the changes were implemented.

3.3.2.2.2 The Contractor shall archive all amendments to the QAPP document for future reference by the Government.

3.3.2.3 The Contractor shall send a copy of the latest version of the QAPP document within 7 days of a written request by the EPA Regional CLP COR or the ASB CLP COR, as directed. The EPA requestor will designate the recipients.

4.0   STANDARD OPERATING PROCEDURES

4.1   Introduction

To obtain reliable results, adherence to prescribed analytical methodology is imperative. In any operation that is performed on a repetitive basis, reproducibility is best accomplished through the use of SOPs. As defined by the EPA, an SOP is a written document which provides directions for the step-by-step execution of an operation, analysis, or action which is commonly accepted as the method for performing certain routine or repetitive tasks. The Contractor shall follow the EPA Guidance for Preparing Standard Operating Procedures (SOPs) (QA/G-6).

4.1.1   SOPs prepared by the Contractor shall be functional (i.e., clear, comprehensive, up to date, and sufficiently detailed to permit duplication of results by qualified analysts).

4.1.2   All SOPs shall reflect activities as they are currently performed in the laboratory. In addition, all SOPs shall be:

- Consistent with current EPA regulations, guidelines, and the CLP contract's requirements;

- Consistent with instrument(s) manufacturer's specific instruction manuals;

- Available to the Government during an on-site laboratory evaluation. A complete set of SOPs shall be bound together and available for inspection at such evaluations. During on-site laboratory evaluations, laboratory personnel may be asked to demonstrate the application of the SOPs;

- Available to designated recipients within 7 days, upon request by the EPA Regional CLP COR or the ASB CLP COR;

- Capable of providing for the development of documentation that is sufficiently complete to record the performance of all tasks required by the protocol;

- Capable of demonstrating the validity of data reported by the Contractor and explaining the cause of missing or inconsistent results;

- Capable of describing the corrective measures and feedback mechanism utilized when analytical results do not meet protocol requirements;

- Reviewed regularly and updated as necessary when contract, facility, or Contractor procedural modifications are made;

- Archived for future reference in usability or evidentiary situations;

- Available at specific workstations, as appropriate;

- Reviewed and signed by all Contractor personnel performing actions identified in the SOP; and

- Subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

4.2   Format

The format for SOPs may vary depending upon the type of activity for which they are prepared. The SOPs shall be paginated consecutively in ascending order. At a minimum, the following sections shall be included:

- Title Page;

- Document Control;

- Scope and Applicability;

- Summary of Method;

- Definitions (acronyms, abbreviations, and specialized forms used in the SOP);

- Health and Safety;

- Personnel Qualifications;

- Interferences;

- Apparatus and Materials (list or specify, also note designated locations where found);

- Handling and Preservation;

- Instrument or Method Calibration;

- Sample Preparation and Analysis;

- Data Calculations;

- Procedures;

- QC limits;

- Corrective action procedures, including procedures for secondary review of information being generated;

- Documentation description and example forms;

- Data Management and Records Management;

- Miscellaneous notes and precautions; and

- References.

4.3   Required Standard Operating Procedures

The Contractor shall maintain the following SOPs:

4.3.1   Evidentiary SOPs for required chain of custody and document control.

4.3.2   Sample receipt and storage:

- Sample receipt and identification logbooks;

- Refrigerator temperature logbooks;

- Extract storage logbooks; and

- Security precautions.

4.3.3   Sample preparation:

- Reagent purity check procedures and documentation;

- Extraction/Digestion/Distillation procedures;

- Extraction/Digestion/Distillation bench sheets; and

- Extraction/Digestion/Distillation logbook maintenance.

4.3.4   Glassware cleaning

4.3.5   Calibration (balances, pipets, etc.):

- Procedures;

- Frequency requirements;

- Preventative maintenance schedule and procedures;

- Acceptance criteria and corrective actions; and

- Logbook maintenance authorization.

4.3.6   Analytical procedures (for each analytical system):

- Instrument performance specifications;

- Instrument operating procedures;

- Data acquisition system operation;

- Procedures used when automatic quantitation algorithms are overridden;

- QC-required parameters;

- Analytical sequence/injection logbooks; and

- Instrument error and editing flag descriptions and resulting corrective actions.

4.3.7   Maintenance activities (for each analytical system):

- Preventative maintenance schedule and procedures;

- Corrective maintenance determinants and procedures; and

- Maintenance authorization.

4.3.8   Analytical standards:

- Standard coding/identification and inventory system;

- Standards preparation logbook(s);

- Standard preparation procedures;

- Procedures for equivalency/traceability analyses and documentation;

- Purity logbook (primary standards and solvents);

- Storage, replacement, and labeling requirements; and

- QC and corrective action measures.

4.3.9   Data reduction procedures:

- Data processing systems operation;

- Outlier identification methods;

- Identification of data requiring corrective action; and

- Procedures for format and/or forms for each operation.

4.3.10   Documentation policy/procedures:

- Contractor/analyst's notebook policy, including review policy;

- CSF contents;

- CSF organization and assembly procedures, including review policy; and

- Document inventory procedures, including review policy.

4.3.11   Data validation/self-inspection procedures:

- Data flow and chain of command for data review;

- Procedures for measuring precision and accuracy;

- Evaluation parameters for identifying systematic errors;

- Procedures to ensure that hardcopy and electronic deliverables are complete and compliant with the requirements in Exhibit B – Reporting and Deliverables Requirements and Exhibit H – Format for Electronic Data Deliverables;

- Procedures to ensure that hardcopy deliverables are in agreement with their comparable electronic deliverables;

- Demonstration of internal QA inspection procedure [demonstrated by supervisory sign-off on personal notebooks, internal Performance Evaluation (PE) samples, etc.];

- Frequency and type of internal audits (e.g., random, quarterly, spot checks, perceived trouble areas);

- Demonstration of problem identification, corrective actions, and resumption of analytical processing. Sequence resulting from internal audit (i.e., QA feedback); and

- Documentation of audit reports (internal and external), response, corrective action, etc.

4.3.12   Data management and handling:

- Procedures for controlling and estimating data entry errors;

- Procedures for reviewing changes to data and deliverables and ensuring traceability of updates;

- Lifecycle management procedures for testing, modifying, and implementing changes to existing computing systems to include hardware, software, and documentation or installation of new systems;

- Database security, backup, and archival procedures including recovery from system failures;

- System maintenance procedures and response time;

- Individual(s) responsible for system operation, maintenance, data integrity, and security;

- Specifications for staff training procedures;

- Virus Protection procedures for software and electronic data deliverables; and

- Storage, retrieval, and verification of the completeness and readability of instrument files transferred to electronic media.

4.4   Submission of the Standard Operating Procedures

4.4.1   Initial Submission

The Contractor is required to submit their SOPs to the EPA CO within 60 days after contract award. The Contractor shall maintain on file a complete set of SOPs, fully compliant with the requirements of this contract for the term of the contract.

4.4.2   Revision Submissions

The revised SOPs will become the official SOPs under the contract and may be used during legal proceedings.

4.4.2.1   During the term of the contract, the Contractor shall amend the SOPs when the following circumstances occur:

- The EPA modifies the technical requirements of the SOW or the contract;

- The EPA notifies the Contractor of deficiencies in their SOP documentation;

- The EPA notifies the Contractor of deficiencies resulting from the EPA's review of the Contractor's performance;

- The Contractor's procedures change;

- The Contractor identifies deficiencies resulting from the internal review of SOP documentation; or

- The Contractor identifies deficiencies resulting from the internal review of procedures.

4.4.2.2   The Contractor shall amend and submit revised or write and submit new SOPs to the recipient(s) identified in Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule, within 14 days of when the circumstances listed above result in a discrepancy between what was previously described in the SOPs, and what is presently occurring at the Contractor's facility.

4.4.2.2.1   All changes in the SOPs shall be clearly marked (e.g., a bar in the margin indicating where the change is in the document, or highlighting the change by underlining the change, bold printing the change, or using a different print font) and the amended/new SOPs shall have the date on which the changes were implemented.

4.4.2.2.2   The Contractor shall document the reasons for the changes and archive all amended SOPs for future reference by the Government. Documentation of the reason(s) for changes to the SOPs shall also be submitted along with the SOPs.

4.4.2.3   The Contractor shall send a copy of the latest version of the SOPs within 7 days of a written request by the EPA Regional CLP COR or the ASB CLP COR, as directed. The EPA requestor will designate the recipients.

5.0   CHAIN OF CUSTODY

5.1   Introduction

A sample is physical evidence collected from a facility or the environment. Controlling evidence is an essential part of the hazardous waste investigation effort. To ensure that the EPA's sample data and records supporting sample-related activities are admissible as evidence in litigation, Contractors are required to maintain EPA furnished samples under chain of custody and to account for all samples and supporting records of sample handling, preparation, and analysis.

The Contractor shall develop and implement the following SOPs for sample chain of custody (COC) under this contract. The Contractor shall provide the following SOPs: sample receiving, sample identification, sample security, sample storage, sample tracking and document control, electronic sample data control, and CSF organization and assembly to ensure accountability of sample chain of custody, as well as control of all sample-related records.

5.2   Sample Receiving

5.2.1   The Contractor shall designate a sample custodian responsible for receiving Government-furnished samples.

5.2.2   The Contractor shall designate a representative to receive Government-furnished samples in the event that the sample custodian is not available.

5.2.3   The sample custodian or a designated representative shall verify and record on Form DC-1 the agreement or disagreement of information recorded on all documents received with samples and information recorded on sample containers.

5.2.4   The sample custodian or a designated representative shall verify and record the following information on Form DC-1 as samples are received and inspected:

- Presence or absence and condition of custody seals on shipping and/or sample containers;

- Custody seal numbers, when present;

- Condition of the sample bottles;

- Presence or absence of airbills or airbill stickers;

- Airbill or airbill sticker numbers;

- Presence or absence of Traffic Report/Chain of Custody (TR/COC) Records;

- Sample tags/numbers listed/not listed on TR/COC Records;

- Presence or absence of shipping container temperature indicator bottle;

- Shipping container temperature;

- Date of receipt;

- Time of receipt;

- EPA Sample Numbers;

- Presence or absence of sample tags;

- Sample tag numbers;

- Assigned laboratory numbers;

- Remarks regarding condition of sample shipment;

- Samples delivered by hand; and

- Problems and discrepancies.

5.2.5   The sample custodian or a designated representative shall sign, date, and record the time on all accompanying forms, when applicable, at the time of sample receipt (e.g., TR/COC Records or packing lists, and airbills).

NOTE: Initials are not acceptable.

5.2.6   The Contractor shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to: absent documents, conflicting information, and absent or broken custody seals.

5.2.7   The Contractor shall record resolution of all problems and discrepancies communicated through SMO.

5.3   Sample Identification

5.3.1   The Contractor shall maintain the identity of Government-furnished samples and prepared samples (including digested samples and distilled samples) throughout the laboratory.

5.3.2   Each sample and sample preparation container shall be labeled with the EPA Sample Number or a unique laboratory sample identification number.

5.4   Sample Security

5.4.1   The Contractor shall demonstrate that sample custody is maintained from receiving through retention or disposal. A sample is in custody if:

- It is in your possession; or

- It is in your view after being in your possession; or

- It is locked in a secure area after being in your possession; or

- It is in a designated secure area, accessible only to authorized personnel.

5.4.2   The Contractor shall demonstrate security of designated secure areas.

5.5   Sample Storage

The Contractor shall designate storage areas for Government-furnished samples and prepared samples.

5.6   Sample Tracking and Document Control

5.6.1   The Contractor shall record all activities performed on Government-furnished samples.

5.6.2   Titles which identify the activities recorded shall be printed on each page of all laboratory documents (activities include, but are not limited to: sample receipt, sample storage, sample preparation, sample analysis, CSF organization and assembly, and sample retention or disposal). When a document is a record of analysis, the instrument type and parameter group shall be included in the title.

5.6.3   When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

5.6.4   Reviewers' signatures shall be identified on laboratory documents when reviews are conducted.

NOTE: Individuals recording review comments on computer-generated raw data are not required to be identified unless the written comments address data validity. The Laboratory Name shall be identified on pre-printed laboratory documents.

5.6.5   Each laboratory document entry shall be dated in the format MM/DD/YYYY (e.g., 01/01/2016) and signed (or initialed) by the individual(s) responsible for performing the recorded activity at the time the activity is recorded.

5.6.6   Notations on laboratory documents shall be recorded in ink.

5.6.7   Corrections to laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.

5.6.8   Unused portions of laboratory documents shall be lined out, signed (or initialed), and dated.

5.6.9   Pages in bound and unbound logbooks shall be sequentially numbered.

5.6.10 Each page in bound and unbound logbooks shall be dated (MM/DD/YYYY) and signed (no initials) at the bottom by the individual recording the activity (if a single entry is made on a page) or by the last individual recording information on the page (if multiple entries are on the same page).

5.6.11 Instrument-specific analytical sequence logs shall be maintained to enable the reconstruction of analytical sequences.

5.6.12 Logbook entries shall be in chronological order.

5.6.13 Information inserted into laboratory documents shall be affixed permanently in place. The individual responsible for inserting information shall sign and date across the insert and logbook page at the time information is inserted.

5.6.14 The Contractor shall document disposal or retention of Government-furnished samples, remaining portions of samples, and prepared samples.

5.7   Electronic Sample Data Control

5.7.1   Contractor personnel responsible for original data entry shall be identified at the time of data input.

5.7.2   The Contractor shall make changes to electronic data in a manner which ensures that the original data entry is preserved, the editor is identified, and the revision date is recorded.

5.7.3   The Contractor shall routinely verify the accuracy of manually entered data, electronically entered data, and data acquired from instruments.

5.7.4   The Contractor shall routinely verify documents produced by the electronic data collection system to ensure accuracy of the information reported.

5.7.5   The Contractor shall ensure that the electronic data collection system is secure.

5.7.5.1   The electronic data collection system shall be maintained in a secure location.

5.7.5.2   Access to the electronic data collection system functions shall be limited to authorized personnel through utilization of software security techniques (e.g., log-ons or restricted passwords).

5.7.5.3   Electronic data collection systems shall be protected from the introduction of external programs or software (e.g., viruses).

5.7.6   The Contractor shall designate archive storage areas for electronic data and the software required to access the data.

5.7.7   The Contractor shall designate an individual responsible for maintaining archives of electronic data, including the software.

5.7.8   The Contractor shall maintain the archives of electronic data and necessary software in a secure location that shall be accessible only to authorized personnel.

5.8   Complete Sample Delivery Group File Organization and Assembly

5.8.1   The Contractor shall designate a Document Control Officer responsible for the organization and assembly of the CSF.

5.8.2   The Contractor shall designate a representative responsible for the organization and assembly of the CSF in the event that the Document Control Officer is not available.

5.8.3   The Contractor shall maintain documents relating to the CSF in a secure location.

5.8.4   All original laboratory forms and copies of SDG-related logbook pages shall be included in the CSF.

5.8.5   Copies of laboratory documents in the CSF shall be photocopied in a manner to provide complete and legible replicates.

5.8.6   Documents relevant to each SDG including, but not limited to, the following shall be included in the CSF:

- Logbook pages;
- Bench sheets;
- Screening records;
- Preparation records;
- Repreparation records;
- PE sample instructions;
- Chromatograms;
- Analytical records;
- Reanalysis/Re-extraction records;
- TR/COC Records;
- Sample tracking records;

- Raw data summaries;
- Computer printouts;
- Records of failed or attempted analysis;
- Correspondence;
- FAX originals; and
- Other.

5.8.7   The Document Control Officer or a designated representative shall ensure that sample tags are encased in clear plastic bags before placing them in the CSF.

5.8.8   CSF documents shall be organized and assembled on an SDG-specific basis.

5.8.9   Original documents which include information relating to more than one SDG (e.g., TR/COC Records, calibration logs) shall be filed in the CSF with the lowest SDG Number, and copies of these originals shall be placed in the other CSF(s). The Document Control Officer or a designated representative shall record the following statement on the copies in (indelible) dark *ink*:

<div align="center">

COPY
ORIGINAL DOCUMENTS ARE INCLUDED IN CSF

</div>

<div align="right">

_____

Signature

_____

Date

</div>

5.8.10 All CSFs shall be submitted with a completed Form DC-2. All resubmitted CSFs shall be submitted with a new or revised Form DC-2.

5.8.11 Each item in the CSF and resubmitted CSFs shall be inventoried and assembled in the order specified on Form DC-2. Each page of the CSF shall be stamped with a sequential number. Page number ranges shall be recorded in the columns provided on Form DC-2. Intentional gaps in the page numbering sequence shall be recorded in the "Comments" section on Form DC-2. When inserting new or inadvertently omitted documents, the Contractor shall identify them with unique accountable numbers. The unique accountable numbers and the locations of the documents shall be recorded in the "Other Records" section on Form DC-2.

5.8.12 Before shipping each CSF, the Document Control Officer or a designated representative shall verify the agreement of information recorded on all documentation and ensure that the information is consistent and the CSF is complete.

5.8.13 The Document Control Officer or a designated representative shall document the shipment of deliverable packages, including what was sent, to whom the packages were sent, the date, and the carrier used.

5.8.14 Shipments of deliverable packages, including resubmittals, shall be sealed with custody seals by the Document Control Officer or a designated representative in a manner such that opening the packages would break the seals.

5.8.15 Custody seals shall be signed and dated by the Document Control Officer or a designated representative when sealing deliverable packages.

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EXHIBIT F

PROGRAMMATIC QUALITY ASSURANCE/QUALITY CONTROL ELEMENTS

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Exhibit F – Programmatic Quality Assurance/Quality Control Elements

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1.0   OVERVIEW

Quality Assurance (QA) and Quality Control (QC) are integral parts of
the U.S. Environmental Protection Agency's (EPA's) Contract Laboratory
Program (CLP). This integrated program is required to generate data of
known and documented quality. The QA process consists of management
reviews and oversight at the planning, implementation, and completion
stages of the environmental data collection activity, and ensures that
data provided are of the quality required. The QC process includes
those activities required during data collection to produce the data
quality desired and to document the quality of the collected data.

During the planning of an environmental data collection program, the
activities focus on defining data quality criteria and designing a QC
system to measure the quality of the data being generated. During the
implementation of the data collection effort, the QA activities ensure
that the QC system is functioning effectively, and the deficiencies
uncovered by the QC system are corrected. After the environmental data
are collected, QA activities focus on assessing the quality of data
obtained to determine it suitability to support enforcement or remedial
decisions.

2.0   INTRODUCTION

Appropriate use of data generated under the large range of analytical
conditions encountered in environmental analyses requires reliance on
the QC procedures and criteria incorporated into the methods. The data
acquired from QC procedures are used to estimate and evaluate the
information content of analytical results and to determine the
necessity for, or the effects of, corrective action procedures. The
parameters used to estimate information content include precision,
accuracy, and other quantitative and qualitative indicators.

This Exhibit describes the overall programmatic QA/QC operations and
the minimum QC operations necessary to satisfy the analytical
requirements associated with the determination of the different method
analytes. These QC operations are designed to facilitate laboratory
comparison by providing the EPA with comparable data from all
Contractors. These requirements do not release the analytical
Contractor from maintaining their own QC checks on method and
instrument performance.

3.0   GENERAL QUALITY ASSURANCE/QUALITY CONTROL PRACTICES

The necessary components of a complete QA/QC program include internal
QC criteria that demonstrate compliant levels of performance, as
determined by the Contractors' QA review and external QC review of data
and procedures that is accomplished by the monitoring activities of the
EPA.

Each external review accomplishes a different purpose. External
reviews may include: Proficiency Testing, contract compliance
screening, on-site laboratory audits, data package audits, electronic
data audits, and the EPA Regional data review. A feedback loop
provides the results of these various review functions to the
Contractor through communications with the EPA.

4.0   PROFICIENCY TESTING PROGRAM

As a means of measuring and evaluating both the Contractor's and the method's analytical performance, the Contractor shall participate in the EPA's Proficiency Testing (PT) Program. The EPA's PT Program involves the analysis of Case-specific Performance Evaluation (PE) samples and PT audits. The Contractor's PE and PT audit sample results will be used by the EPA to assess and verify the Contractor's continuing ability to produce acceptable analytical data in accordance with the contractual requirements. The Contractor must receive a passing score of 75% to be in compliance with the contract.

4.1   Performance Evaluation Samples

4.1.1   PE sample(s) may be scheduled with the Contractor as frequently as on a Sample Delivery Group (SDG)-by-SDG basis.

4.1.2   PE samples will be provided as either single-blinds (recognizable as a PE sample, but of unknown composition), or as double-blinds (not recognizable as a PE sample and of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PE samples.

4.1.3   The Contractor may receive the PE samples as either full volume samples or ampulated/bottled concentrates from the EPA or a designated EPA Contractor. The PE samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PE samples (i.e., the required dilution of the PE sample concentrate). PE samples are to be extracted and analyzed with the rest of the routine samples in the SDG. The Contractor shall prepare and analyze the PE sample using the procedure described in the sample preparation and method analysis sections of Exhibit D – Analytical Methods. All contract required QC shall also be met.

4.1.4   The PE sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B – Reporting and Deliverables Requirements. If these requirements are not met, the EPA Region may reject all the data associated with the SDG.

4.1.5   The Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PE sample. When PE sample results are received by the EPA, the PE sample results will be evaluated for correct analytical identification and quantitation. The results of the PE sample evaluation will be provided to the Contractor via coded evaluation sheets, by analyte. The EPA will notify the Contractor of unacceptable performance.

4.2   Proficiency Testing Audits

4.2.1   A PT audit is a unique analytical Case containing only PT audit samples. The PT audit samples will be scheduled by the EPA Analytical Services Branch (ASB) through the Sample Management Office (SMO). PT audit samples assist the EPA in monitoring Contractor performance.

4.2.2   PT audit samples will be provided as single-blinds (recognizable as a PT audit sample but of unknown composition). The Contractor will not be informed of either the analytes or the concentrations in the PT audit samples.

4.2.3   The Contractor may receive the PT audit samples as either full volume samples or ampulated/bottled concentrates from the EPA or a designated EPA Contractor. The PT audit samples shall come with instructions concerning the unique preparation procedures, if any, required to reconstitute the PT audit samples (i.e., the required dilution of the PT audit sample concentrate). The Contractor shall prepare and analyze the PT audit samples using the procedure described in the sample preparation and method analysis sections of Exhibit D – Analytical Methods. All contract required QC shall be met, including spike and duplicate.

4.2.4   The PT audit sample results are to be submitted in the SDG deliverable package per normal reporting procedures detailed in Exhibit B – Reporting and Deliverables Requirements.

4.2.5   The Contractor shall be responsible for correctly identifying and quantitating the analytes included in each PT audit sample. When PT audit sample results are received by the EPA, the PT audit sample results will be scored for correct analytical identification, quantitation, and timeliness. The PT audit sample scoring will be provided to the Contractor via coded evaluation sheets, by analyte.

4.2.6   The EPA will notify the Contractor of unacceptable performance. The Contractor's overall and method-specific PT audit sample performance will be assessed into one of the following three categories:

4.2.6.1   Acceptable, No Response Required: Score greater than or equal to 90%. The data meets most or all of the scoring criteria. No response is required.

4.2.6.2   Acceptable, Response Explaining Deficiencies Required: Score greater than or equal to 75%, but less than 90%. Deficiencies exist in the Contractor's performance. Corrective action response required.

4.2.6.3   Unacceptable Performance, Response Explaining Deficiencies Required: Score less than 75%. Corrective action response required.

4.2.7   In the case of Section 4.2.6.2 or 4.2.6.3, the Contractor shall describe the deficiency(ies) and the action(s) taken in a corrective action letter to the EPA Contracting Officer (CO), the EPA Regional CLP Contracting Officer's Representative (COR), and the ASB CLP COR, within 14 days of receipt of notification from the EPA.

4.2.8   A remedial PT audit is a unique analytical Case containing only PT audit samples. A remedial PT audit may be scheduled by EPA ASB with the Contractor(s) for any of the following reasons: unacceptable PE sample performance and/or major change in the laboratory (e.g., relocation, new owner, or high turnover of key personnel). The Contractor may not receive samples under this contract until acceptable performance of a remedial PT audit sample is achieved. Sections 4.2.2 through 4.2.7 apply to the remedial PT audit process.

4.2.9   The Contractor shall be notified by the EPA CO concerning agreement or disagreement with the proposed remedy for unacceptable performance.

5.0   CONTRACT COMPLIANCE SCREENING

5.1   Overview

5.1.1   Contract Compliance Screening (CCS) is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the Complete SDG File (CSF) delivered to the EPA.

5.1.2   CCS is performed by SMO at the direction of the EPA. To ensure uniform review, a set of standardized procedures has been developed to evaluate the CSF submitted by a Contractor against the technical and completeness requirements of the contract. The EPA reserves the right to add and/or delete individual checks.

5.2   Contract Compliance Screening Results

CCS results are distributed to the Contractor and all other data recipients. The Contractor shall correct deficiencies and submit corrections within 6 business days. The Contractor shall send all corrections to the EPA Regional CLP COR and SMO. CCS results are used in conjunction with other information to measure overall Contractor performance and to take appropriate actions to correct deficiencies in performance.

5.3   Contract Compliance Screening Trend Report

The EPA will periodically generate a CCS Trend Report which summarizes CCS results over a given period of time. The Government may send the CCS Trend Report to the Contractor, or discuss the CCS Trend Report during an on-site laboratory audit. In a detailed letter to the EPA Regional CLP COR, the ASB CLP COR, and the EPA CO, the Contractor shall address the deficiencies and the subsequent corrective actions implemented by the Contractor to correct the deficiencies within 14 days of receipt of the report.

6.0   ON-SITE LABORATORY AUDITS

6.1   Overview

The EPA Regional CLP COR, the ASB CLP COR, or the EPA CO's authorized representative will conduct an on-site laboratory audit. On-site laboratory audits are performed to monitor the Contractor's ability to meet selected terms and conditions specified in the contract.

6.2   On-Site Audit

QA evaluators inspect the Contractor's facilities to verify the adequacy and maintenance of instrumentation; the continuity, experience and education of personnel; and the acceptable performance of analytical and QC procedures. Auditors conduct on-site laboratory audits to evaluate if laboratory policies and procedures are in place to satisfy evidence handling requirements.

6.2.1   The items to be monitored during an on-site audit may include, but not be limited to, the following:

- Size and appearance (e.g., cleanliness, organization) of the facility;

- Quantity, age, availability, scheduled maintenance, and performance of instrumentation;

- Availability, review, appropriateness, and utilization of the Quality Assurance Project Plan (QAPP) and Standard Operating Procedures (SOPs);

- Staff qualifications, experience, and personnel training programs;

- Analysis of PE samples (may be in the presence of the EPA-designated team);

- Reagents, standards, and sample storage facilities;

- All logbooks (e.g., digestion logs, standards and reagent preparation logs, analysis logs, instrument maintenance logs);

- All raw analytical data; and

- Review of the Contractor's sample analysis, data package assembly, inspection, completion, and data management procedures.

6.2.2   Prior to an on-site audit, various documentation pertaining to performance of the Contractor is reviewed by the audit team and may be discussed during the audit. Items that may be discussed include, but not limited to, the following:

- Previous on-site audit reports;

- PE or PT audit sample scores;

- EPA Regional review of data;

- Contractor performance information;

- Data and Electronic audit reports;

- Results of CCS; and

- Data trend reports.

6.3   Discussion of the On-Site Audit Findings

The auditors shall present their findings and recommendations for corrective actions necessary to the Contractor personnel during a debriefing meeting at the conclusion of the audit. A report which discusses deficiencies found during the on-site audit will be sent to the Contractor to provide further clarification of findings.

6.3.1   In a detailed letter to the EPA Regional CLP COR, the ASB CLP COR, and the EPA CO, the Contractor shall discuss the deficiencies and the subsequent corrective actions implemented by the Contractor to resolve the deficiencies within 14 days of receipt of report.

7.0   DATA PACKAGE AUDITS

7.1   Overview

Audits provide the EPA with an in-depth inspection and evaluation of the Case data package with regard to achieving QA/QC acceptability. Data package audits enable the EPA to evaluate the implementation, precision, and accuracy of the analytical methods. The audits are performed by the EPA to support the following activities:

- Program overview;

- Contractual requirements and data consistency;

- Identification/Investigation of data quality problems;

- Support for on-site laboratory audits; and

- Specific EPA Regional requests.

7.2   Required Information

Data packages are periodically selected from recently received Cases and evaluated for the technical quality of hardcopy raw data, QA, and the adherence to contractual requirements. A thorough review of the raw data is completed, including all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements; a check for transcription and calculation errors; a review of the qualifications of the laboratory personnel involved with the Case; and a review of the latest version of all SOPs on file. This function provides external monitoring of program QC requirements. Data package audits are used to assess the technical quality of the data and evaluate overall laboratory performance.

7.3   Submission Request

The data package from a recent Case, a specific Case, or a PE sample may be requested. Upon request from the EPA Regional CLP COR, the ASB CLP COR, or the EPA CO, the Contractor shall send the required data package and all necessary documentation to the EPA designated recipient within 7 days of notification in accordance with Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

7.4   Response to the Data Package Audit Report

After completion of the data package audit, the EPA shall make the data package audit report available to the Contractor. In a detailed letter to the designated recipients, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the data package audit report within 14 days of receipt of the report.

8.0   ELECTRONIC DATA AUDITS

8.1   Overview

Audits provide the EPA with an in-depth inspection and evaluation of the electronic data with regard to achieving QA/QC acceptability. Electronic data audits enable the EPA to evaluate the implementation, precision, and accuracy of the analytical methods. The audits are performed by the EPA to support the following activities:

- Program overview;

- Contractual requirements and data consistency;

- Identification/Investigation of data quality problems;

- Support for on-site laboratory audits; and

- Specific EPA Regional requests.

8.2   Required Information

Data packages are periodically selected from recently received Cases and evaluated for the technical quality of hardcopy raw data, QA, and the adherence to contractual requirements. A thorough review of the raw data is completed, including all instrument readouts used for the sample results, instrument printouts, and other documentation for deviations from the contractual requirements; a check for transcription and calculation errors; a review of the qualifications of the laboratory personnel involved with the Case; and a review of the latest version of all SOPs on file. This function provides external monitoring of program QC requirements. Electronic data audits are used to assess the technical quality of the data and evaluate overall laboratory performance.

8.2.1   The Contractor shall store all raw and processed analytical data in appropriate instrument manufacturer's proprietary software format uncompressed and with no security codes. This data shall include all necessary data files for a complete reconstruction of the previously submitted hardcopy and electronic deliverable data package. The Contractor is required to retain the instrument electronic data for 3 years after submission of the reconciled CSF.

8.2.2   All associated raw data files in the instrument manufacturer proprietary software format must be submitted if those files contain data or instrumental parameters regarding any analysis and or correction applied to an instrument or analytical result. This electronic data shall include data for all samples, blanks, Laboratory Control Samples (LCSs), matrix spikes, post-digestion/distillation spikes, duplicates, serial dilutions, Interference Check Samples, tunes, initial calibrations/verifications, and continuing calibration verifications.

8.2.3   The Contractor shall maintain a written reference logbook of data files of the EPA Sample Number, calibration data, standards, spikes, duplicates, and blanks. The logbook shall include the EPA Sample Numbers and standard and blank IDs, identified by Case.

8.2.4   The Contractor shall supply upon request raw data for the Method Detection Limit (MDL) studies which are used to set the MDL values for the SDG.

8.2.5   Electronic data shipped to the EPA-designated recipient must be fully usable by the recipient. When submitting instrument electronic data to the EPA, the following materials shall be delivered in response to the request:

8.2.5.1   All associated raw data files for all analytical samples, calibration, and QC data.

8.2.5.1.1   Instrument data files for Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) shall include raw intensities and as applicable, associated background corrected and background subtracted intensities. Mercury and Cyanide files shall include raw absorbances or integrated areas.

8.2.5.2   All processed data files and quantitation output files associated with the raw data files described in Section 8.2.5.1.

8.2.5.3   All associated identification and calculation files used to generate the data submitted in the data package. This includes, but is not limited to: result files, acquisition files, calibration files, and method files.

8.2.5.4   References relating data files to EPA Sample Numbers, calibration data, standards, blanks, spikes, duplicates, and LCSs. The logbook shall include the EPA Sample Numbers and Lab File Identifiers for all samples, blanks, and standards, identified by Case and SDG.

8.2.5.5   A printout of the directory of all files in each directory, including all subdirectories and the files contained therein.

8.2.5.6   A copy of the CSF, if an audit request is made within the period during which the Contractor must retain a copy.

8.2.5.7   A statement attesting to the completeness of the instrument electronic data submission, signed and dated by the Contractor's Laboratory Manager or Manager's designee. The Contractor shall also provide a statement attesting that the data reported have not been altered in any way. These statements shall be part of a cover sheet that includes the following information relevant to the data file submission:

- Contractor name;

- Date of submission;

- Case Number;

- SDG Number;

- Instrument manufacturer and model number;

- Instrument operating software and version number;

- Data system computer;

- System operating software;

- Data system network;

- Data backup software/service;

- Data analysis software;

- Media type and volume of data (in MB) backed up; and

- Names and telephone numbers of two Contractor contacts for further information regarding the submission.

8.3   Submission of Request

The instrument electronic data from a recent Case, a specific Case, or a PE sample may be requested. Upon request from the EPA Regional CLP COR, the ASB CLP COR, or the EPA CO, the Contractor shall send the required instrument electronic data and all necessary documentation to the EPA designated recipient within 7 days of notification in accordance with Exhibit B – Reporting and Deliverables Requirements, Table 1 – Deliverable Schedule.

8.4   Response to the Electronic Data Audit Report

After completion of the electronic data audit, the EPA will make the electronic data audit report available to the Contractor. In a detailed letter to the designated recipients, the Contractor shall discuss the corrective actions implemented to resolve the deficiencies listed in the electronic data audit report within 14 days of receipt of the report.

9.0   REGIONAL DATA REVIEW

9.1   Overview

Contractor data are generated to meet the specific needs of the EPA Regions. In order to verify the usability of data for the intended purpose, each EPA Region reviews data from the perspective of the end user, based on functional guidelines for data review, which have been developed jointly by the EPA Regions and EPA ASB. Each EPA Region uses the guidelines as the basis for data evaluation. Individual EPA Regions may augment the basic guideline review process with additional review based on the EPA Region-specific or site-specific concerns. The EPA Regional reviews, like the sites under investigation, vary based on the nature of the problem under investigation and the EPA Regional response appropriate to the specific circumstances.

The EPA Regional data reviews, relating usability of the data to a specific site, are part of the collective assessment process. They complement the review done by SMO, which is designed to identify contractual discrepancies, and the review done by EPA ASB, which is designed to evaluate Contractor and method performance.

9.2   Submission Request

As part of the CLP contractual requirements, CLP laboratories shall deliver their CSF for each SDG to the EPA Region where the samples have been collected. The EPA Regional recipients are identified at the time of scheduling. The data shall be shipped in accordance to the procedures described in Exhibit B – Reporting and Deliverables Requirements of this Statement of Work (SOW). The EPA Regions use the hardcopy data to perform their data review. The EPA Region may contact the laboratory after they initiate or complete their review requesting additional information or clarification. The Contractor shall respond to the request within 5 business days (exception 3 days for a 7-day turnaround).

10.0 TABLES

TABLE 1. Contract Laboratory Program Quality Assurance Monitoring Plan

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit A: Summary of Requirements	Summary of Program Requirements	Performance standards are summarized in Exhibit A, Sections 1.0 through 4.0.	QA monitoring plan is outlined in Exhibit F.
Exhibit B: Reporting and Deliverables Requirements	Reporting and Deliverable Requirements	Performance standards are outlined in Exhibit B, Sections 1.0 through 4.0.	CCS in Exhibit F, Section 5.0, and SMO data review will be used to monitor reporting electronic deliverables.
Exhibit C: Inorganic Target Analyte List and Contract Required Quantitation Limits	Target Analyte List and Contract Required Quantitation Limits	Performance standards are outlined in Exhibit C.	QA monitoring plan is outlined in Exhibit F.
Exhibit D: Inorganic Analytical Methods	Introduction to Analytical Methods	Performance standards for stock standards are outlined in Exhibit D, Introduction, Section 4.0, and must be performed as stated.	Randomly, the EPA will review analytical standards verification and preparation documentation, as deemed appropriate.
	General Inorganic Analyses requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	ICP-AES requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	ICP-MS requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit D: Inorganic Analytical Methods (Cont'd)	Mercury requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
	Cyanide requirements are outlined in Exhibit D, Sections 1.0 through 8.0, 14.0, and 15.0.	Performance standards are outlined in Exhibit D, Sections 9.0 through 12.0.	QA monitoring plan is outlined in Exhibit D, Section 12.0, and Exhibit F.
Exhibit E: Quality Systems	General QA/QC Requirements	As outlined in each Exhibit D, Section 12.0.	QA Management Plan is outlined in Exhibit E, Section 2.0.
	Quality Assurance Project Plan	As outlined in Exhibit E, Section 3.0, a written QAPP shall be used to ensure acceptable data production of known and documented quality.	The EPA will review and approve the QAPP after contract award and throughout the contract term as needed. *[The Quality Management Plan (QMP) will be reviewed and approved by the EPA pre contract award.]*
	Standard Operating Procedures	Performance standards are outlined in Exhibit E, Section 4.0, and must be performed as stated.	SOPs will be reviewed by the EPA during on-site audits, after modifications are made, and randomly, as deemed appropriate.
	Data Management	Performance standards are outlined in Exhibit E, Section 4.3.12.	The EPA will monitor data management practices during quality assurance and evidentiary on-site audits.

SOW Reference	Performance Requirements	Performance Standards	QA Monitoring Plan
Exhibit F: Programmatic Quality Assurance/ Quality Control Elements	Proficiency Audit Testing	Performance standards are outlined in Exhibit F, Section 4.0, and must be performed as stated.	Acceptable PT audit scores will assist in monitoring Contractor performance as defined in Exhibit F, Section 4.2.5.
	Contract Compliance Screening	Performance standards are outlined in the contract and must be performed as stated.	CSF will be evaluated against the technical and completeness requirements of the contract.
	On-Site Laboratory Audits	Performance standards are outlined in Exhibit F, Section 6.2.	The EPA will evaluate the results from quality assurance and evidentiary on-site audits as defined in Exhibit F, Section 6.3, to assist in monitoring the Contractor.
	Data Package Audits	Performance standards are outlined in Exhibit F, Section 7.0.	Data package audits are performed by the EPA to evaluate technical quality of the hardcopy raw data, QA, and adherence to contractual requirements.
	Electronic Data Evaluation and Audits	Performance standards are outlined in Exhibit F, Section 8.0.	The EPA uses Exhibit F, Section 8.0, to monitor laboratory electronic deliverables.
	Regional Data Review	Analytical data is reviewed by each Region from the perspective of the end user to determine the usability of the data, as outlined in Exhibit F, Section 9.0.	The EPA Regional validation and/or SMO data review reports are generated for all data packages.
Exhibit G: Glossary of Terms	Glossary of Terms	Contractors shall adhere to interpretation of SOW terms as defined within Exhibit G.	N/A
Exhibit H: Format for Electronic Data Deliverables	Data Dictionary and Format	Performance standards are outlined in Exhibit H.	CCS in Exhibit F, Section 5.0, will be used to monitor electronic deliverables.

EXHIBIT G

GLOSSARY OF TERMS

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ABSORBANCE - A measure of the decrease in incident light passing through a sample into a detector. It is defined mathematically as:

$$A = -\log \frac{I}{I_O}$$

WHERE, I = Radiation intensity of a sample.
             I_O = Radiation intensity of a blank.

ALIQUOT - A measured portion of a field sample, standard, or solution taken for sample preparation and/or analysis.

ANALYSIS DATE/TIME - The date and military time (24-hour clock) of the introduction of the sample, standard, or blank into the analysis system.

ANALYTE - The element or ion an analysis seeks to determine; the element of interest.

ANALYTICAL METHOD - Specifies the procedures for sample preparation, instrument calibration, sample analysis, and result calculations.

ANALYTICAL REFERENCE STANDARD - Standards purchased from private chemical supply companies used to prepare calibration standards and Continuing Calibration Verification (CCV) standards.

ANALYTICAL SAMPLE - Any solution or media introduced into an instrument on which an analysis is performed, excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), Continuing Calibration Blank (CCB), and tunes. Note the following are all defined as analytical samples: undiluted and diluted samples (EPA and non-EPA), matrix spike samples, duplicate samples, serial dilution samples, post-digestion spike samples, Interference Check Samples (ICSs), Laboratory Control Samples (LCSs), Performance Evaluation (PE) samples, and Preparation Blanks.

ANALYTICAL SEQUENCE - The order of actual instrumental analysis of the samples, from the time of instrument calibration through the analysis of the final Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB). All sample analyses during the analytical sequence are subject to the Quality Control (QC) protocols set forth in Exhibit D – Analytical Methods and Exhibit F – Programmatic Quality Assurance/Quality Control Elements of the contract, unless otherwise specified in the individual methods.

ANALYTICAL SERVICES BRANCH (ASB) - The division of the United States Environmental Protection Agency's (EPA's) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP).

ASTM/ASTM INTERNATIONAL – A developer and provider of voluntary consensus standards.

BACKGROUND CORRECTION - A technique to compensate for variable background contribution to the instrument signal in the determination of trace elements.

BATCH - A group of samples prepared at the same time in the same location using the same method.

BLANK - An analytical sample that has negligible or unmeasurable amounts of a substance of interest. The blank is designed to assess specific sources of contamination. Types of blanks may include calibration blanks, preparation blanks, and field blanks. See the individual definitions for types of blanks.

CALIBRATED MASS - 1) A mass whose apparent mass has been adjusted from the uncalibrated mass by the instrumental mass calibration software routine. 2) An analyte mass whose intensity counts have been calibrated against standards of known analyte concentration.

CALIBRATION - A set of operations that establish under specific conditions, the relationship between values indicated by a measuring instrument and the corresponding known values. The calibration standards must be prepared using the same type of reagents or concentration of acids as used in the sample preparation.

CALIBRATION BLANK - A blank solution containing all of the reagents and in the same concentration as those used in the analytical sample preparation. This blank is not subjected to the preparation method for Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), but is digested for mercury and cyanide. Calibration blanks are used to verify that the instrument baseline is stable and the instrument is free of contamination.

CALIBRATION STANDARDS - A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the calibration curve). The solutions may or may not be subjected to the preparation method but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.

CASE - A finite, usually predetermined number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

CLASS A GLASSWARE – Defined by ASTM standards as glassware used in measurement with the smallest degree of uncertainty or tolerance associated with a measurement of volume.

CONTAMINATION - A component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

CONTINUING CALIBRATION VERIFICATION (CCV) - A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be analyzed at the beginning of the day prior to the analysis of samples, and every 2 hours (1 hour for Hg and CN).

CONTRACT COMPLIANCE SCREENING (CCS) - A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is done under EPA direction by the SMO Contractor.

CONTRACT LABORATORY PROGRAM (CLP) - Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technology Innovation (OSRTI) of the EPA.

CONTRACT REQUIRED QUANTITATION LIMIT (CRQL) - Minimum level of quantitation acceptable under the contract Statement of Work (SOW), and supported by the analysis of standards.

CONTROL LIMITS - A range within which specified measurement results must fall to be compliant. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that noncompliant data be flagged.

CYANIDE (Total) - Cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

DATE - The date format for all reporting forms is MM/DD/YYYY - Where MM = 01 for January, 02 for February, ... 12 for December; DD = 01 to 31; YYYY = 2015, 2016, etc.

DAY - Unless otherwise specified, day shall mean calendar day.

DIGESTION LOG - An official record of the sample preparation (digestion or distillation).

DISSOLVED METALS - Analyte elements in an aqueous/water sample which will pass through a 0.45 micrometer (µm) filter.

DRY WEIGHT - The weight of a sample based on percent solids. The weight after drying in an oven.

DUPLICATE - A second aliquot of a sample that is treated the same as the original sample in order to evaluate the precision.

EPA ASB INORGANIC CLP CONTRACTING OFFICER'S REPRESENTATIVE (ASB CLP COR) - The EPA ASB official who manages the Inorganic CLP Program.

EPA CONTRACTING OFFICER (CO) - The EPA official who has the authority to enter into, administer, terminate contracts, and/or make related determinations and findings.

EPA REGIONAL CLP CONTRACTING OFFICER'S REPRESENTATIVE (REGIONAL CLP COR) - The EPA official who monitors assigned CLP laboratories (either inside or outside of the Regional CLP COR's respective Region), responds to and identifies problems in laboratory operations, and participants in on-site laboratory audits.

EPA SAMPLE NUMBER - A unique identification number designated by the EPA for each sample. The EPA Sample Number appears on the Sample Traffic Report/Chain of Custody Record which documents information on that sample.

FIELD BLANK - Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

FIELD QC - Any Quality Control (QC) samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, field duplicates, and field spikes.

FIELD SAMPLE - A portion of material received to be analyzed that is contained in single or multiple containers and identified by a unique EPA Sample Number.

FORM – A hardcopy and/or electronic information/data entry sheet with locked preformatted structure that guides and/or controls user entry/input.

HARDNESS (TOTAL) – Total hardness is defined as the sum of calcium and magnesium concentrations, both expressed as calcium carbonate in micrograms/Liter (mg/L). Total hardness is calculated according to the Standard Method 2340B.

HOLDING TIME - Contractual holding time is the elapsed time expressed in days from the date of receipt of the sample by the Contractor until the date of its analysis.

Holding time = (sample analysis date – sample receipt date)

INDEPENDENT STANDARD - A Contractor-prepared standard solution that is composed of analytes from a different source than those used in the standards for the calibration.

INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROSCOPY (ICP-AES) – A technique for the simultaneous or sequential multi-element determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Characteristic atomic line emission spectra are produced by excitation of the sample in a radio frequency inductively coupled plasma.

INDUCTIVELY COUPLED PLASMA - MASS SPECTROMETRY (ICP-MS) - A technique for the multi-element determination of elements in solution. The basis of the technique is the detection of atomic ions produced by an ICP and sorted by mass-to-charge (m/z) ratio.

IN-HOUSE - At the Contractor's facility.

INITIAL CALIBRATION - Analysis of analytical standards for a series of different concentrations; used to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

INITIAL CALIBRATION VERIFICATION (ICV) - Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when the EPA ICV solutions are not available.

INTERFERENCE CHECK SAMPLE (ICS) - A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors.

INTERFERENTS - Substances which affect the analysis for the analyte of interest.

INTERNAL STANDARD - A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

LABORATORY - Synonymous with Contractor, as used herein.

LABORATORY CONTROL SAMPLE (LCS) - A reference matrix spiked with target analytes at known concentrations. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

LABORATORY RECEIPT DATE - The date on which a sample is received at the Contractor's facility, as recorded on the shipper's delivery receipt and Sample Traffic Report/Chain of Custody Record. Also referred to as Validated Time of Sample Receipt (VTSR).

MATRIX - The predominant material of which the sample to be analyzed is composed. For the purpose of this Statement of Work (SOW), a sample matrix is either aqueous/water, soil/sediment, or a wipe. Matrix is not synonymous with phase (liquid or solid).

MATRIX EFFECT – In general, the effect of a particular matrix on the constituents under study. The enhancement or suppression of minor element spectral lines due to a particular matrix constituent.

MATRIX SPIKE - Aliquot of a sample (aqueous/water or soil/sediment) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure to indicate the appropriateness of the method for the matrix by measuring recovery.

METHOD DETECTION LIMIT (MDL) – The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.

MONITORED MASS - A mass that counts are collected from during analysis that may be subsequently used in isobaric correction equations or for the interpretation of possible interferences in analyte mass results.

PERCENT DIFFERENCE (%D) - The difference between the two values divided by one of the values multiplied by 100.

PERCENT RECOVERY (%R) – The percentage of an analyte or element added to a sample that is recovered. It is the difference between the concentration detected in the spiked sample and that detected in the original (unspiked) sample, divided by the concentration added to the spiked sample multiplied by 100.

PERCENT SOLIDS (%S) - The proportion of solid in a soil/sediment sample determined by drying an aliquot of the sample.

PERFORMANCE EVALUATION (PE) SAMPLE - A sample of known composition to the EPA; however, unknown to the Contractor that is provided to evaluate Contractor performance.

POST-DIGESTION SPIKE – Post-digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post-digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

PREPARATION BLANK - An analyte-free sample to which all reagents are added in the same volume or proportions as used in sample processing. The preparation blank must be carried through the entire sample preparation and analytical procedures. It is used to assess contamination resulting from the analytical process.

PREPARATION LOG - An official record of the sample preparation (digestion or distillation).

PROFICIENCY TESTING (PT) AUDIT SAMPLE – A sample of known composition provided by the EPA for Contractor analysis. Used by the EPA to evaluate Contractor performance on a program-wide basis.

QUALITY ASSURANCE TECHNICAL SUPPORT (QATS) LABORATORY - A Contractor-operated facility operated under the QATS contract, awarded and administered by the EPA.

RAW DATA - The originally recorded and unprocessed measurements from any measuring device such as analytical instruments, balances, pipettes, thermometers, etc.

REAGENT WATER - The purity of this water must be equivalent to ASTM Type II reagent water of Specification D1193-06, "Standard Specification for Reagent Water".

REFERENCE MATERIAL - Standards, typically provided by the EPA, used to verify method and instrument performance. Examples include Initial Calibration Verification (ICV) standards and Interference Check Solution (ICS) standards.

RELATIVE PERCENT DIFFERENCE (RPD) - The relative percent difference is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

REPORTED DATA - Reported data are processed from the raw measurement values that may have been reformatted from the original measurement to meet specific reporting requirements, such as significant figures and decimal precision.

ROUNDING RULES - If the figure following those to be retained is greater than or equal to 5, round up; otherwise, round down. As an example, 11.443 is rounded down to 11.4 and 11.455 is rounded up to 11.5. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures. See specific form instructions (Exhibit B – Reporting and Deliverables Requirements) for exceptions.

SAMPLE - A portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

SAMPLE DELIVERY GROUP (SDG) - A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or

- Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

- All samples scheduled with the same level of deliverables.

- In addition, all samples assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.

Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory. Laboratories shall take all precautions to meet the 20 sample per SDG criteria.

SAMPLE MANAGEMENT OFFICE (SMO) - A Contractor-operated facility operated under the SMO contract, awarded and administered by the EPA.

SDG NARRATIVE - Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution. Complete Sample Delivery Group (SDG) Narrative specifications are included in Exhibit B – Reporting and Deliverables Requirements.

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

SOIL - Synonymous with soil/sediment as used herein.

STOCK SOLUTION - A standard solution which can be diluted to derive other standards.

SUPPORTING DATA – Any data that substantiates the Reported Data (see definition above), including initial instrument measurements, instrument result calculations, standards concentrations, standard concentration calculations, sample preparation data (e.g., initial/final sample volume measurements, reagent quantities, etc.), Method Detection Limits (MDLs), and Interelement Corrections (IECs). Supporting data include standard preparation logs, sample preparation logs, instrument analysis logs, MDL and IEC studies, balance logs, pipette logs, percent solids logs, etc.

TARGET ANALYTE LIST - A list of Inorganic Analytes (metals and cyanide) as designated in Exhibit C – Inorganic Target Analyte List and Contract Required Quantitation Limits.

TIME - hh:mm:ss - When required to record time on any deliverable item, time shall be expressed as Military Time [i.e., a 24-hour clock (0000-2359)].

TRAFFIC REPORT/CHAIN OF CUSTODY RECORD (TR/COC) - An EPA sample
identification form completed by the sampler, which accompanies the sample
during shipment to the laboratory and is used to document sample identity,
sample chain of custody, sample condition, and sample receipt by the
laboratory.

TUNE - A solution containing a range of isotope masses to establish
Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) accuracy, resolution,
and precision prior to calibration. May also be called Instrument
Performance Check sample (IPC).

VALIDATED TIME OF SAMPLE RECEIPT (VTSR) - The date on which a sample is
received at the Contractor's facility, as recorded on the shipper's delivery
receipt and sample Traffic Report/Chain of Custody Record.

EXHIBIT H

FORMAT FOR ELECTRONIC DATA DELIVERABLES

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Exhibit H – Format for Electronic Data Deliverables

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1.0   INTRODUCTION

The inorganic analytical service provides analytical data for use by the U.S. Environmental Protection Agency (EPA), in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). The electronic data deliverable (EDD) requirements in this section are designed to allow the EPA and other federal agencies or programs to rapidly assess the accuracy, completeness, and usefulness of the analytical results and the data. Depending on the stage chosen, the data user will receive results, support quality control (QC), and verification of the calculated results and quality measures.

2.0   FORMAT CHARACTERISTICS

2.1   This constitutes an implementation of the Staged Electronic Data Deliverable (SEDD) based on analytical results and other associated information required by the contract. Because this implementation is specific to the contract, not all data elements listed in the cross-program Document Type Definition (DTD) are required. This implementation is based on SEDD Specification 5.2 that can be found at:

https://www.epa.gov/clp/staged-electronic-data-deliverable-sedd

2.1.1   The SEDD deliverable consists of an eXtensible Markup Language (XML) file(s) compliant with the XML specification 1.0 of the World Wide Web Consortium (W3C). The deliverable must be well-formed based on the W3C XML specification and must be valid based on the DTD.

2.1.2   The Contractor shall create the deliverable using the UTF-8 (Unicode Transformation Format - 8 bit) character set.

2.1.3   The EDD SEDD stage delivery level (2a, 2b, or 3) must match the EPA requested/scheduled EDD SEDD level.

2.1.4   The initial line of the deliverable shall be: <?xml version="1.0" encoding="UTF-8"?>.

2.1.5   The second line of the deliverable shall be a DOCTYPE line that contains the filename of the DTD. The DOCTYPE line shall be <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_3_3.dtd">, <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_2b_3.dtd">, or <!DOCTYPE Header SYSTEM "SEDD_5-2_GENERAL_2a_2.dtd">, where "Header" denotes the name of the root element, and "SEDD_5-2_GENERAL_3_3.dtd" (for a Level 3 deliverable), "SEDD_5-2_GENERAL_2b_3.dtd" (for a Level 2b deliverable), or "SEDD_5-2_GENERAL_2a_2.dtd" (for a Level 2a deliverable) denotes the filename of the DTD.

2.1.6   The use of XML comment lines is permitted at any position in the file after the first two lines.

2.2   This implementation includes detailed specifications for the required
        format of the content of each data element for each analytical method.
        The content of each data element is specified as either literal
        (contained in quotes) which must appear exactly as shown (without
        quotes), or as a variable for which descriptions and formats are
        listed. Exhibit H, Section 3.0 describes the requirements for each
        data element.

2.2.1   For this implementation, numeric data elements may contain numeric
        digits, a decimal place, and a leading minus sign. Values without a
        leading minus sign are assumed to be positive. Values must be
        reported to the specified precision or significance.

2.2.2   The values reported by the Contractor are used for data assessment.
        No raw data values in the SEDD files shall be rounded. The
        Contractor shall not use rounded intermediate values in calculating
        the final result, and no rounding shall be performed until reaching
        the final result.

2.2.3   The completeness of analytical data provided in the EDD will be
        verified against the analytical data requested on the Traffic
        Report/Chain of Custody (TR/COC) Record. The Laboratory Code, Case
        Number, Contract Number, Sample Delivery Group (SDG) Number,
        Modified Analysis (MA) Number (if applicable), sample number, and
        analytical method shall be identical in the EDD and the TR/COC
        Record and the SDG coversheet submitted by the Contractor for the
        SDG.

2.2.4   The following variables shall be present where required and correct:
        EDD Implementation Identifier (ID); Lab ID; Lab Receipt Date;
        Analysis Date and Time; Collected Date; Matrix ID; Client Method ID;
        Client Method Type; QC Type; Instrument ID; Correlation Coefficient
        (level 2b and 3 only); Intercept (level 2b and 3 only); Method ID;
        Run Batch (level 2b and 3 only); Analysis Batch (level 2b and 3
        only); Analysis Group ID (level 2b and 3 only); Client Analysis ID;
        Client Analyte ID; Preparation Batch; Percent Recovery (%R);
        Relative Percent Difference (RPD); Percent Difference (%D); and
        Percent Relative Standard Deviation (%RSD).

3.0   DATA ELEMENTS

3.1   The SEDD consists of data elements arranged hierarchically by data nodes (parent elements). Figures 1, 2, and 3 depict the data node hierarchy. Each data element consists of a start tag, content, and an end tag. An element may contain other elements (child elements).

   NOTE: There shall be no more than one occurrence of each child element within a node, unless the child element also behaves as a parent element. For example, in each SamplePlusMethod node, there may be only one occurrence of the element ClientSampleID, but there may be more than one occurrence of the element Analysis.

   The tags, nodes, and hierarchy are specified in the DTD against which the deliverable will be validated (see Exhibit H, Section 6.0). The frequency requirements for each of the data nodes applicable to this implementation are described below.

3.1.1   Header Node (Required for All Deliverable Levels)

   One Header node must be reported for each analytical method.

3.1.2   SamplePlusMethod Node (Required for All Deliverable Levels)

   Each Header node must contain one SamplePlusMethod node for each field sample, field blank (including rinse, equipment, and trip blanks), Performance Evaluation (PE) sample, Proficiency Testing (PT) audit sample, Matrix Spike (MS) sample, Post-Digestion Spike (PDS) sample (if applicable), Duplicate (Dup) sample, Serial Dilution (SD) sample, Preparation Blank (PB), Leachate Extraction Blank (LEB), Laboratory Control Sample (LCS), and Non-Client Sample (NCS).

3.1.3   ReportedResult Node (Required for All Deliverables Levels)

   Each SamplePlusMethod node must contain one and only one ReportedResult node for each target analyte.

3.1.4   ContactInformation Node (Required for All Deliverable Levels)

   Each Header node must contain one ContactInformation node.

3.1.5   InstrumentQC Node (Required for Levels 2b and 3 Deliverables Only)

   Each Header node must contain one InstrumentQC node for each instrument performance check (ICP-MS Tune), initial calibration sequence, Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Interference Check Samples (ICSA and ICSAB).

3.1.6   AnalysisGroup Node (Required for Levels 2b and 3 Deliverables Only)

   Each initial calibration InstrumentQC node for multi-point calibration must contain one AnalysisGroup node containing summary data for the initial calibration. Each AnalysisGroup node must contain one Analyte node for each target analyte.

3.1.7   Analysis Node (Required for All Deliverable Levels)

   Each SamplePlusMethod node must contain at least one Analysis node. A separate Analysis node is required for each dilution or reanalysis. Each InstrumentQC node (other than Initial Calibration) must contain one Analysis node. Any reanalysis must be preceded by an initial analysis. Any analysis reported as a dilution must also have a less-diluted analysis reported as initial. The initial analysis does not have to precede the diluted analysis.

3.1.8   Analyte Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain one Analyte node for each target analyte (except Hardness), monitored interferent, and internal standard. Each Analysis node under an InstrumentQC node must contain one Analyte node for each target analyte (except Hardness), monitored interferent, and internal standard. Each Analysis node under an InstrumentQC node for tune must contain one Analyte node for each tune analyte. Each AnalysisGroup node must contain one Analyte node for each target analyte.

3.1.9   PreparationPlusCleanup Node (Required for All Deliverable Levels)

Each Analysis node under a SamplePlusMethod node must contain at least one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation". For Serial Dilution and Post-Digestion Spike samples, the associated PreparationPlusCleanup node shall contain data for the preparation of the original sample. For those methods requiring digested QC, each InstrumentQC node must contain one PreparationPlusCleanup node with a PreparationPlusCleanupType equal to "Preparation".

3.1.10   Peak Node (Required for Levels 3 and 2b Deliverables Only)

Each Analyte node must contain at least one Peak node. For Level 2b, only the Analyte nodes under InstrumentQC must contain a Peak node. Within a RunBatch, a peak must be consistently identified. For an Inductively Coupled Plasma – Mass Spectrometer (ICP-MS) using collision or reaction cells on an analyte-by-analyte basis, internal standards reported from collision/reaction cell mode shall be reported with a "-Gas" suffix. If an internal standard applies to both the collision/reaction cell path and the normal path target analytes, report the internal standard results as separate peaks, using the "-Gas" suffix in PeakID to distinguish the collision/reaction cell results from the normal path results.

3.1.11   PeakComparison Node (Required for Levels 2b and 3 Deliverables Only)

For ICP-MS, each Peak node must contain a PeakComparison node for each applicable internal standard.

3.1.12   PeakReplicate Node (Required for Level 3 Deliverables only)

For Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) and ICP-MS, each Peak node must contain a PeakReplicate node for each replicate exposure or integration collected, and shall contain at least the number of PeakReplicate nodes necessary to report the required minimum number of exposures or integrations.

3.1.13   Characteristic Node (Required for All Deliverable Levels)

Each SamplePlusMethod, PreparationPlusCleanup, and Handling node may contain one or more Characteristic nodes, one for each sample characteristic that must be reported for a sample at time of receipt, after preparation, or after handling.

3.1.14   Handling Node (required for Level 3 Deliverables only)

Each SamplePlusMethod node shall contain one or more Handling nodes when Toxicity Characteristic Leaching Procedure (TCLP) and or Synthetic Precipitation Leaching Procedure (SPLP) extraction has been performed.

3.1.15　AnalyteComparison Node (For Level 3 Deliverables only)

For ICP-AES, each Analyte node must contain one AnalyteComparison node for each applicable Interelement Correction Factor.

3.1.16　AnalyteGroup Node

For ICP-AES, each Analysis node under a SamplePlusMethod node must contain one AnalyteGroup node for each derived analyte (i.e., Hardness) when required.

3.2　Detailed instructions for the content of each data element are provided in Tables 1, 2, and 3 of Section 7.0. The following is an explanation of the data fields contained in each table.

3.2.1　Node and Data Elements

This field reports each node in bold text, followed by its data elements. If an entire node is not required, then none of its data elements are listed.

3.2.2　Applicability

This field reports the samples, blanks, and standards for which each node and data element is required. An "X" in a column indicates that the node or element is required. Sample refers to field samples, field blanks, and PE samples unless otherwise noted. Abbreviations used in this field are defined in Section 7.0, Table 4 - Abbreviations.

3.2.3　Instructions

This field describes the required format and content of each data element. The content of each data element is specified as either literal (contained in quotes), or as a variable for which description and format is listed. Abbreviations used in this field are defined in Section 7.0, Table 4 - Abbreviations.

Figure 1: Data Node Hierarchy for
Level 2a Deliverable
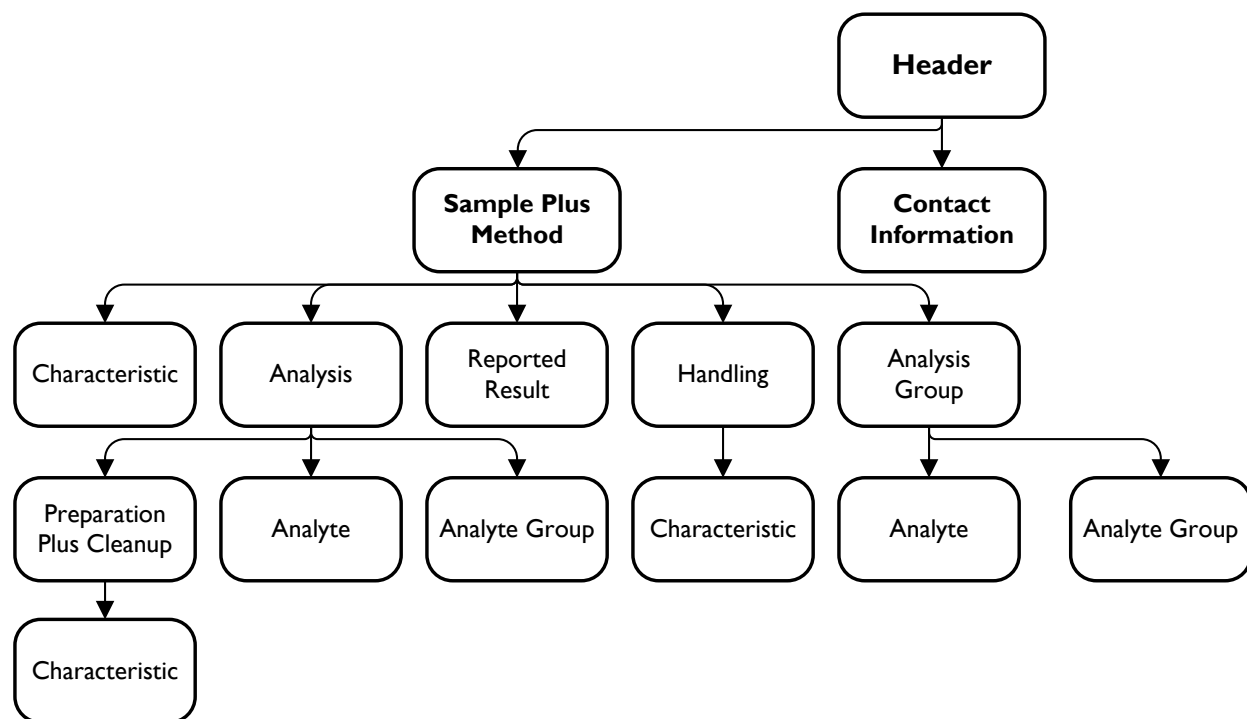
Figure 2: Data Node Hierarchy for
Level 2b Deliverable
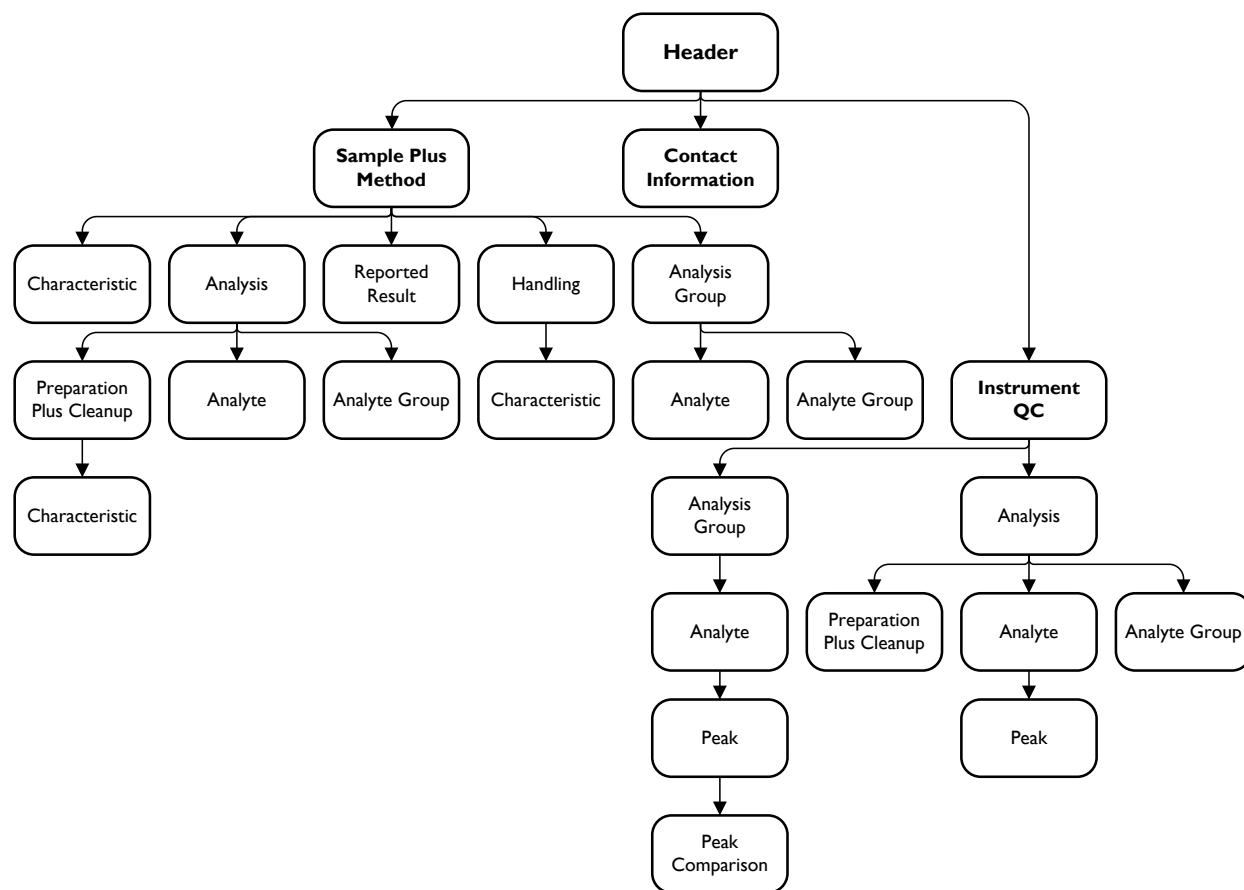
Figure 3: Data Node Hierarchy for
Level 3 Deliverable
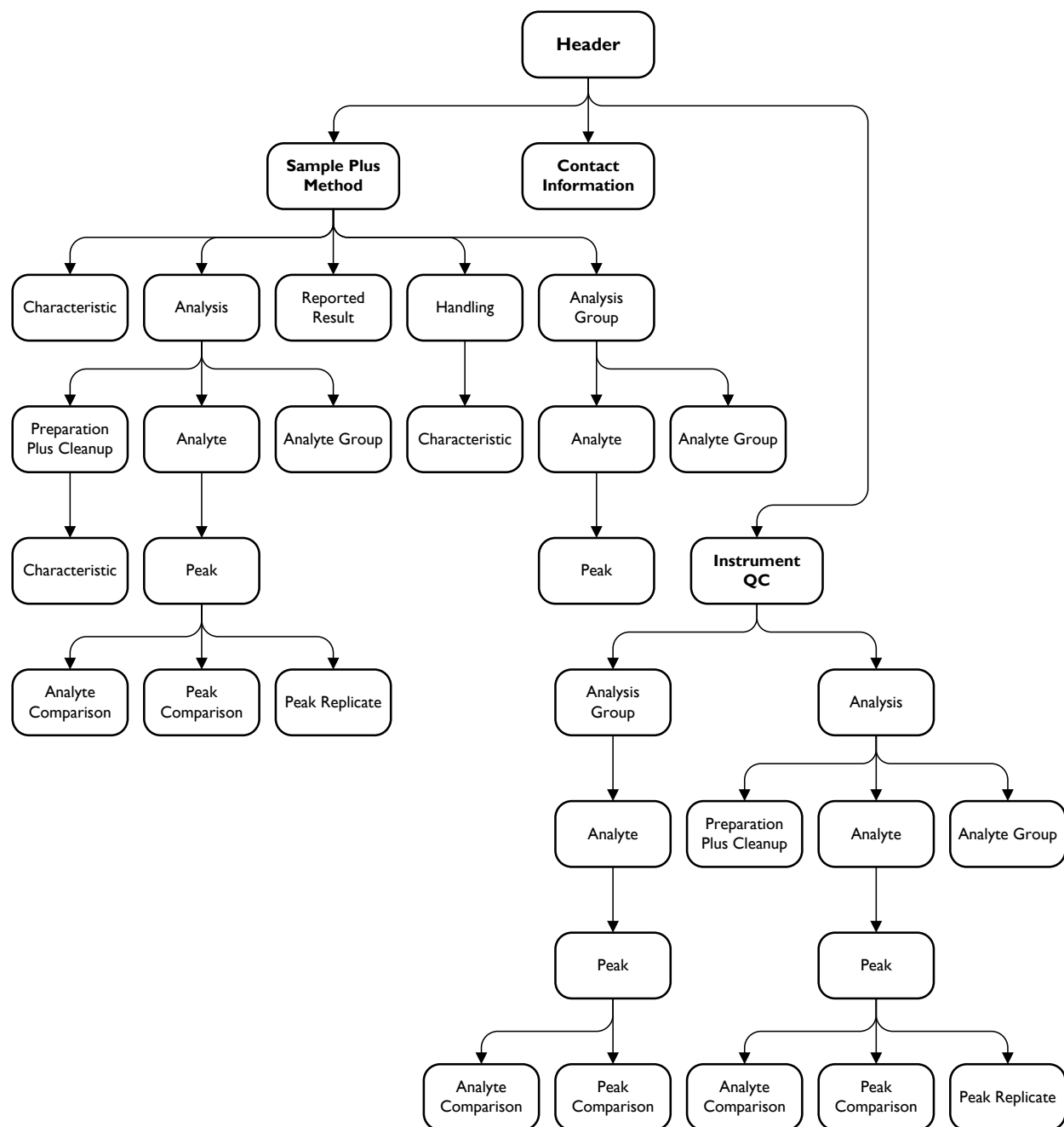
4.0   BATCHES

4.1   This implementation requires the use of the following SEDD batches from
      the SEDD Specification: "LabReportingBatch"; "RunBatch";
      "AnalysisBatch"; and "PreparationBatch". "HandlingBatch" is required
      when TCLP or SPLP extraction is performed.

4.1.1   The "LabReportingBatch" links all samples reported in the same SDG.
        Report the SDG Number.

4.1.2   The "RunBatch" links all analyses performed under the same initial
        calibration. All analyses performed under an initial calibration
        must have the same content for the "RunBatch" element as the initial
        calibration from which their results are calculated.

4.1.3   The "AnalysisBatch" and "AnalysisBatchEnd" link all analyses
        performed within the same analytical sequence (1- or 2-hour period
        and QC). All analyses performed within the same analytical sequence
        must have the same content for the "AnalysisBatch" element as the
        tune or standard(s) that began the analytical sequence, and the same
        content for the "AnalysisBatchEnd" as the standard(s) that ends the
        analytical sequence.

4.1.4   The "PreparationBatch" links all samples of the same matrix prepared
        at the same time by the same preparation method. All samples
        analyzed, including Preparation Blanks, Matrix Spikes, Duplicates,
        and LCSs, that are prepared together must have the same content for
        the "PreparationBatch" element. For those methods using
        digested/distilled QC, all QC that are prepared together must have
        the same content for the "PreparationBatch" element.

4.1.5   The "HandlingBatch" links all samples subjected to TCLP or SPLP
        extraction at the same time by the same method. All samples
        extracted, including the LEB, that are extracted together must have
        the same content for the "HandlingBatch" element.

5.0   DELIVERABLE

5.1   Each analytical method in an SDG shall be submitted as a separate file.
        The Contractor may choose to deliver their file as a ZIP of an XML
        file. For reporting requirements, the analytical methods are:
        "ICP_AES"; "ICP_MS"; "Hg"; and "CN". All analytical methods within an
        SDG shall be submitted at the same time (i.e., the file for the second
        analytical method in an SDG shall be submitted in a single file upload
        with the first analytical method).

5.2   The Contractor will utilize the Electronic Data Exchange and Evaluation
        System (EXES) at https://epasmoweb.fedcsc.com to electronically submit
        their EDD to the Sample Management Office (SMO). The EPA may approve
        alternative electronic means of file delivery. Written permission must
        be obtained from the EPA Analytical Services Branch (ASB) prior to the
        use of any alternative means.

5.3   The Contractor must follow the delivery instructions in Exhibit B –
        Reporting and Deliverables Requirements, of this Statement of Work
        (SOW), and deliver their hardcopy and EDD and Portable Document Format
        (PDF) of the Complete SDG File (CSF) to SMO concurrently. If one of
        these items is delivered on a later date, the Data Receipt Date (DRD)
        for the SDG will be the later of the two dates.

5.4   Information in the electronic deliverable must correspond to
        information submitted in the hardcopy raw data package and on QC
        summary forms. If information in the raw data or on the forms is
        changed, the information in the electronic deliverable shall be changed
        accordingly. An electronic deliverable containing the changed
        information for the SDG shall be resubmitted along with the hardcopy at
        no additional cost to the EPA.

5.5   The format for the file name shall be Case number_SDG number_contract
        number_submission number_DTD used_Method. For example, the first
        submission of the ICP-AES Analytical Method from SDG number MABC12,
        Case number 12345, contract EP-W-00-000 would be named 12345_MABC12_EP-
        W-00000_1_SEDD_5-2_GENERAL_3_3_ICP_AES.zip.

6.0   DOCUMENT TYPE DEFINITION

6.1   Introduction

    The deliverable will be validated against DTD SEDD_5-2_GENERAL_3_3, DTD
    SEDD_5-2_GENERAL_2b_3 or DTD SEDD_5-2_GENERAL_2a_2. The deliverable
    must not contain any tags not included in the DTD and must conform to
    the hierarchical structure modeled in the DTD.

6.2   General Stage 3 DTD

```
<?xml version="1.0" encoding="UTF-8"?>
<!-- SEDD_5-2_GENERAL_3_3.dtd 10/22/2009 -->
<!-- Acronym Description -->
<!-- Coeff - Coefficient -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!-- RRF   - Relative Response Factor -->
<!-- RSD   - Relative Standard Deviation -->
<!ELEMENT Header (
            ClientID|
            ClientName|
            Comment|
            DateFormat|
            EDDID|
            EDDImplementationID|
            EDDImplementationVersion|
            EDDVersion|
            GeneratingSystemID|
            GeneratingSystemVersion|
            LabContract|
            LabContractModificationDescription|
            LabContractModificationID|
            LabDataPackageID|
            LabDataPackageName|
            LabDataPackageVersion|
            LabID|
            LabName|
            LabNarrative|
            LabQualifiersDefinition|
            LabReportedDate|
            ProjectID|
            ProjectName|
            SiteID|
            SiteName|
            ContactInformation|
            SamplePlusMethod|
            InstrumentQC
                    )*>
<!ELEMENT Analysis (
            AliquotAmount|
            AliquotAmountUnits|
            AnalysisBatch|
            AnalysisBatchEnd|
            AnalysisDuration|
```

```
AnalysisDurationUnits|
AnalysisGroupID|
AnalysisType|
Analyst|
AnalyzedAmount|
AnalyzedAmountUnits|
AnalyzedDate|
BackgroundCorrection|
BackgroundRawData|
BackgroundType|
BottleID|
ClientAnalysisID|
ClientMethodCode|
ClientMethodID|
ClientMethodModificationDescription|
ClientMethodModificationID|
ClientMethodName|
ClientMethodSource|
ClientMethodVersion|
Column|
ColumnInternalDiameter|
ColumnInternalDiameterUnits|
ColumnLength|
ColumnLengthUnits|
Comment|
ConfirmationAnalysisID|
Counts|
CountsUncertainty|
CountsUncertaintyConfidenceLevel|
CountsUncertaintyDetermination|
CountsUncertaintyIntervalType|
CountsUncertaintyLimitHigh|
CountsUncertaintyLimitLow|
CountsUncertaintyType|
CountsUnits|
DetectorID|
DetectorType|
DilutionFactor|
Efficiency|
HeatedPurge|
Inclusion|
InjectionVolume|
InjectionVolumeUnits|
InstrumentID|
InterelementCorrection|
LabAnalysisID|
LabFileID|
LabID|
LabMethodID|
LabMethodName|
LabName|
MethodCode|
MethodID|
MethodModificationDescription|
MethodModificationID|
MethodName|
MethodSource|
MethodVersion|
OriginalLabAnalysisID|
```

```
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                RunBatch|
                SampleAmount|
                SampleAmountUnits|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT AnalysisGroup (
                AnalysisGroupID|
                AnalysisType|
                Comment|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT Analyte (
                AmountAdded|
                AmountAddedUnits|
                AmountAddedLocation|
                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                BiasErrorRatio|
                CalibrationBasis|
                CalibrationFactor|
                CalibrationFactorUnits|
                CalibrationType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Coeffa0|
                Coeffa1|
                Coeffa2|
                Coeffa3|
                CoeffOfDetermination|
                CoeffOfDeterminationLimitLow|
                CoeffOfDeterminationLimitType|
                Comment|
                CorrelationCoeff|
                CorrelationCoeffLimitLow|
                CorrelationCoeffLimitType|
                Counts|
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                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
```

```
DetectionLimit|
DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
Efficiency|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType|
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
Inclusion|
IntermediateResult|
IntermediateResultLimitHigh|
IntermediateResultLimitLow|
IntermediateResultLimitType|
IntermediateResultUnits|
LabAnalyteID|
LabQualifiers|
LotNumber|
Mass|
MassLimitHigh|
MassLimitLow|
MassLimitType|
MassUnits|
MeanCalibrationFactor|
MeanCalibrationFactorUnits|
MeanRRF|
MeanRRFLimitLow|
MeanRRFLimitType|
PeakID|
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PercentBreakdownLimitHigh|
PercentBreakdownLimitType|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentMatch|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
PercentRSD|
PercentRSDLimitHigh|
PercentRSDLimitLow|
PercentRSDLimitType|
QuantitationBasis|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
```

```
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                    ResponseLimitHigh|
                    ResponseLimitLow|
                    ResponseLimitType|
                    ResponseUnits|
                    Result|
                    ResultLimitHigh|
                    ResultLimitLow|
                    ResultLimitType|
                    ResultType|
                    ResultUncertainty|
                    ResultUncertaintyConfidenceLevel|
                    ResultUncertaintyDetermination|
                    ResultUncertaintyIntervalType|
                    ResultUncertaintyLimitHigh|
                    ResultUncertaintyLimitLow|
                    ResultUncertaintyType|
                    ResultUncertaintyUnits|
                    ResultUnits|
                    RPD|
                    RPDLimitHigh|
                    RPDLimitType|
                    RPDType|
                    RRF|
                    RRFLimitLow|
                    RRFLimitType|
                    StandardConcentration|
                    StandardConcentrationUnits|
                    StandardDeviation|
                    StandardDeviationUnits|
                    StandardFinalAmount|
                    StandardFinalAmountUnits|
                    StandardID|
                    StandardSource|
                    TailingFactor|
                    TailingFactorLimitHigh|
                    TailingFactorLimitType|
                    Wavelength|
                    WavelengthUnits|
                    WeightingFactor|
                    Peak
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<!ELEMENT AnalyteComparison (
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                    ClientAnalyteID|
                    ClientAnalyteName|
                    Comment|
                    CorrectionFactor|
                    LabAnalyteID
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<!ELEMENT Characteristic (
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                    CharacteristicValue|
                    CharacteristicUnits|
                    Comment
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```

```
<!ELEMENT AnalyteGroup (
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                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                CASRegistryNumber|
                ClientAnalyteID|
                ClientAnalyteName|
                Comment|
                LabAnalyteID|
                LabQualifiers|
                Result|
                ResultType|
                ResultUncertainty|
                ResultUnits
                        )*>
<!ELEMENT ContactInformation (
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                LabAddress2|
                LabCity|
                LabCountry|
                LabID|
                LabName|
                LabPointOfContact|
                LabPointOfContactElectronicAddress|
                LabPointOfContactTitle|
                LabPointOfContactType|
                LabState|
                LabTelephoneNumber|
                LabType|
                LabZipCode
                        )*>
<!ELEMENT Handling (
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                ClientMethodID|
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                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                HandledDate|
                HandlingBatch|
                HandlingType|
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                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                ProcedureID|
                ProcedureName|
```

```
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                    Characteristic
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<!ELEMENT InstrumentQC (
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                    ClientMethodModificationID|
                    ClientMethodName|
                    ClientMethodSource|
                    ClientMethodVersion|
                    Comment|
                    LabID|
                    LabInstrumentQCID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    QCLinkage|
                    QCType|
                    AnalysisGroup|
                    Analysis
                            )*>
<!ELEMENT Peak (
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                    Coeffa1|
                    Coeffa2|
                    Coeffa3|
                    CoeffOfDetermination|
                    CoeffOfDeterminationLimitLow|
                    CoeffOfDeterminationLimitType|
                    Comment|
                    CorrelationCoeff|
                    CorrelationCoeffLimitLow|
                    CorrelationCoeffLimitType|
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                    DetectionLimitType|
                    DetectionLimitUnits|
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                    Efficiency|
                    Inclusion|
                    IntermediateResult|
                    IntermediateResultLimitHigh|
                    IntermediateResultLimitLow|
                    IntermediateResultLimitType|
                    IntermediateResultUnits|
                    LabQualifiers|
                    ManualIntegration|
```

```
Mass|
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MassLimitLow|
MassLimitType|
MassUnits|
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MeanCalibrationFactorUnits|
MeanRetentionTime|
MeanRetentionTimeLimitHigh|
MeanRetentionTimeLimitLow|
MeanRetentionTimeLimitType|
MeanRetentionTimeUnits|
MeanRRF|
MeanRRFLimitLow|
MeanRRFLimitType|
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PeakRatio|
PeakRatioLimitHigh|
PeakRatioLimitLow|
PeakRatioLimitType|
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PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRatio|
PercentRatioLimitHigh|
PercentRatioLimitLow|
PercentRatioLimitType|
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PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
PercentRSD|
PercentRSDLimitHigh|
PercentRSDLimitLow|
PercentRSDLimitType|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Resolution|
ResolutionLimitHigh|
ResolutionLimitLow|
ResolutionLimitType|
ResolutionType|
ResolutionUnits|
Response|
ResponseLimitHigh|
ResponseLimitLow|
ResponseLimitType|
ResponseType|
ResponseUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
```

```
                    ResultLimitType|
                    ResultType|
                    ResultUncertainty|
                    ResultUnits|
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                    RetentionTimeLimitHigh|
                    RetentionTimeLimitLow|
                    RetentionTimeLimitType|
                    RetentionTimeUnits|
                    RRF|
                    RRFLimitLow|
                    RRFLimitType|
                    StandardDeviation|
                    StandardDeviationUnits|
                    TailingFactor|
                    TailingFactorLimitHigh|
                    TailingFactorLimitType|
                    Wavelength|
                    WavelengthUnits|
                    WeightingFactor|
                    AnalyteComparison|
                    PeakComparison|
                    PeakReplicate
                            )*>
<!ELEMENT PeakComparison (
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                    ClientAnalyteID|
                    ClientAnalyteName|
                    Comment|
                    LabAnalyteID|
                    PeakID|
                    PeakRatio|
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                    PercentRatio|
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                    PercentRatioLimitLow|
                    PercentRatioLimitType
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<!ELEMENT PeakReplicate (
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                    IntermediateResultLimitType|
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                    MassLimitHigh|
                    MassLimitLow|
                    MassLimitType|
                    MassUnits|
                    PeakReplicateID|
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                    ResolutionLimitHigh|
                    ResolutionLimitLow|
                    ResolutionLimitType|
```

```
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                ResponseLimitLow|
                ResponseLimitType|
                ResponseType|
                ResponseUnits
                        )*>
<!ELEMENT PreparationPlusCleanup (
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                AliquotAmountUnits|
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                CleanupBatch|
                CleanupType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Comment|
                Efficiency|
                FinalAmount|
                FinalAmountUnits|
                InitialAmount|
                InitialAmountUnits|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LotNumber|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                PreparationPlusCleanupType|
                PreparationType|
                PreparedDate|
                ProcedureID|
                ProcedureName|
                SampleAmount|
                SampleAmountUnits|
                Solvent|
                Characteristic
                        )*>
<!ELEMENT ReportedResult (
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                AnalyteGroupID|
                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
```

```
BiasErrorRatio|
CASRegistryNumber|
ClientAnalyteID|
ClientAnalyteName|
ClientDetectionLimit|
ClientDetectionLimitUnits|
ClientQuantitationLimit|
ClientQuantitationLimitUnits|
Comment|
DetectionLimit|
DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType|
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
LabAnalysisID|
LabAnalyteID|
LabQualifiers|
LabResultStatus|
PeakID|
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PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
ResultLimitType|
ResultType|
ResultUncertainty|
ResultUncertaintyConfidenceLevel|
ResultUncertaintyDetermination|
ResultUncertaintyIntervalType|
ResultUncertaintyLimitHigh|
ResultUncertaintyLimitLow|
ResultUncertaintyType|
ResultUncertaintyUnits|
ResultUnits|
RetentionTime|
RetentionTimeUnits|
```

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                    RPDType
                            )*>
<!ELEMENT SamplePlusMethod (
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                    BottleType|
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                    ClientMethodID|
                    ClientMethodModificationDescription|
                    ClientMethodModificationID|
                    ClientMethodName|
                    ClientMethodSource|
                    ClientMethodType|
                    ClientMethodVersion|
                    ClientName|
                    ClientSampleID|
                    CollectedDate|
                    CollectedEndDate|
                    Comment|
                    Composite|
                    CoolerID|
                    CustodyID|
                    EquipmentBatch|
                    Filtered|
                    LabContract|
                    LabContractModificationID|
                    LabContractModificationDescription|
                    LabID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    LabReceiptDate|
                    LabReportingBatch|
                    LabSampleID|
                    LocationID|
                    LocationName|
                    MatrixID|
                    MatrixMedium|
                    MethodBatch|
                    MethodCategory|
                    MethodCode|
                    MethodID|
                    MethodLevel|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodType|
                    MethodVersion|
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                    OriginalLabSampleID|
                    PhaseAnalyzed|
                    Preservative|
                    ProjectID|
                    ProjectName|
                    QCCategory|
```

```
                    QCLinkage|
                    QCType|
                    Quarantine|
                    SamplingBatch|
                    ShippingBatch|
                    SiteID|
                    SiteName|
                    StorageBatch|
                    Analysis|
                    ReportedResult|
                    Handling|
                    AnalysisGroup|
                    Characteristic
                              )*>
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<!ELEMENT Wavelength (#PCDATA)>
<!ELEMENT WavelengthUnits (#PCDATA)>
<!ELEMENT WeightingFactor (#PCDATA)>
<!ELEMENT Yield (#PCDATA)>
```

6.3   General Stage 2b DTD

```
<?xml version="1.0" encoding="UTF_8"?>
<!—SEDD_5-2_GENERAL_2b_3.dtd 10/22/2009 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- Coeff - Coefficient -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!-- RRF   - Relative Response Factor -->
<!-- RSD   - Relative Standard Deviation -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod|
                InstrumentQC
                        )*>
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                AnalysisBatchEnd|
                AnalysisDuration|
                AnalysisDurationUnits|
                AnalysisGroupID|
                AnalysisType|
                Analyst|
                AnalyzedAmount|
                AnalyzedAmountUnits|
                AnalyzedDate|
                ClientAnalysisID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
```

```
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                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
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                ColumnInternalDiameter|
                ColumnInternalDiameterUnits|
                ColumnLength|
                ColumnLengthUnits|
                Comment|
                ConfirmationAnalysisID|
                Counts|
                CountsUncertainty|
                CountsUncertaintyConfidenceLevel|
                CountsUncertaintyDetermination|
                CountsUncertaintyIntervalType|
                CountsUncertaintyLimitHigh|
                CountsUncertaintyLimitLow|
                CountsUncertaintyType|
                CountsUnits|
                DetectorID|
                DetectorType|
                DilutionFactor|
                Efficiency|
                HeatedPurge|
                Inclusion|
                InjectionVolume|
                InjectionVolumeUnits|
                InstrumentID|
                LabAnalysisID|
                LabFileID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                MethodCode|
                MethodID|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodVersion|
                PreparationBatch|
                ProcedureID|
                ProcedureName|
                ReferenceDate|
                ResultBasis|
                RunBatch|
                Temperature|
                TemperatureUnits|
                Wavelength|
                WavelengthUnits|
                Yield|
                PreparationPlusCleanup|
                Analyte|
                AnalyteGroup
                        )*>
<!ELEMENT AnalysisGroup (
                AnalysisGroupID|
                AnalysisType|
                Comment|
```

```
                    Analyte|
                    AnalyteGroup
                            )*>
<!ELEMENT Analyte (
                    AnalyteGroupID|
                    AnalyteName|
                    AnalyteNameContext|
                    AnalyteType|
                    BiasErrorRatio|
                    CalibrationBasis|
                    CalibrationFactor|
                    CalibrationFactorUnits|
                    CalibrationType|
                    CASRegistryNumber|
                    ClientAnalyteID|
                    ClientAnalyteName|
                    Coeffa0|
                    Coeffa1|
                    Coeffa2|
                    Coeffa3|
                    CoeffOfDetermination|
                    CoeffOfDeterminationLimitLow|
                    CoeffOfDeterminationLimitType|
                    Comment|
                    CorrelationCoeff|
                    CorrelationCoeffLimitLow|
                    CorrelationCoeffLimitType|
                    Counts|
                    CountsUncertainty|
                    CountsUncertaintyConfidenceLevel|
                    CountsUncertaintyDetermination|
                    CountsUncertaintyIntervalType|
                    CountsUncertaintyLimitHigh|
                    CountsUncertaintyLimitLow|
                    CountsUncertaintyType|
                    CountsUnits|
                    DetectionLimit|
                    DetectionLimitType|
                    DetectionLimitUnits|
                    DifferenceErrorRatio|
                    Efficiency|
                    ExpectedResult|
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                    ExpectedResultUncertaintyConfidenceLevel|
                    ExpectedResultUncertaintyDetermination|
                    ExpectedResultUncertaintyIntervalType|
                    ExpectedResultUncertaintyLimitHigh|
                    ExpectedResultUncertaintyLimitLow|
                    ExpectedResultUncertaintyType|
                    ExpectedResultUncertaintyUnits|
                    ExpectedResultUnits|
                    Inclusion|
                    LabAnalyteID|
                    LabQualifiers|
                    LotNumber|
                    Mass|
                    MassUnits|
                    MeanCalibrationFactor|
                    MeanCalibrationFactorUnits|
                    MeanRRF|
                    MeanRRFLimitLow|
```

```
                MeanRRFLimitType|
                PeakID|
                PercentBreakdown|
                PercentBreakdownLimitHigh|
                PercentBreakdownLimitType|
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                PercentDifferenceLimitHigh|
                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
                PercentRecovery|
                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                PercentRSD|
                PercentRSDLimitHigh|
                PercentRSDLimitLow|
                PercentRSDLimitType|
                QuantitationBasis|
                QuantitationLimit|
                QuantitationLimitType|
                QuantitationLimitUnits|
                ReportingLimit|
                ReportingLimitType|
                ReportingLimitUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUncertaintyConfidenceLevel|
                ResultUncertaintyDetermination|
                ResultUncertaintyIntervalType|
                ResultUncertaintyLimitHigh|
                ResultUncertaintyLimitLow|
                ResultUncertaintyType|
                ResultUncertaintyUnits|
                ResultUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType|
                RRF|
                RRFLimitLow|
                RRFLimitType|
                StandardSource|
                TailingFactor|
                TailingFactorLimitHigh|
                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                Peak
                        )*>
<!ELEMENT AnalyteGroup (
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                AnalyteName|
                AnalyteNameContext|
                AnalyteType|
                CASRegistryNumber|
```

```
                    ClientAnalyteID|
                    ClientAnalyteName|
                    Comment|
                    LabAnalyteID|
                    LabQualifiers|
                    Result|
                    ResultType|
                    ResultUncertainty|
                    ResultUnits
                              )*>
<!ELEMENT Characteristic (
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                    CharacteristicValue|
                    CharacteristicUnits|
                    Comment
                              )*>
<!ELEMENT ContactInformation (
                    LabAddress1|
                    LabAddress2|
                    LabCity|
                    LabCountry|
                    LabID|
                    LabName|
                    LabPointOfContact|
                    LabPointOfContactElectronicAddress|
                    LabPointOfContactTitle|
                    LabPointOfContactType|
                    LabState|
                    LabTelephoneNumber|
                    LabType|
                    LabZipCode
                              )*>
<!ELEMENT Handling (
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                    ClientMethodID|
                    ClientMethodModificationDescription|
                    ClientMethodModificationID|
                    ClientMethodName|
                    ClientMethodSource|
                    ClientMethodVersion|
                    Comment|
                    HandledDate|
                    HandlingBatch|
                    HandlingType|
                    InitialAmount|
                    InitialAmountUnits|
                    LabID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    ProcedureID|
                    ProcedureName|
                    SampleAmount|
```

```
                    SampleAmountUnits|
                    Characteristic
                            )*>
<!ELEMENT InstrumentQC (
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                    ClientMethodCode|
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                    ClientMethodModificationID|
                    ClientMethodName|
                    ClientMethodSource|
                    ClientMethodVersion|
                    Comment|
                    LabID|
                    LabInstrumentQCID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    QCLinkage|
                    QCType|
                    AnalysisGroup|
                    Analysis
                            )*>
<!ELEMENT Peak (
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                    CalibrationFactorUnits|
                    CalibrationType|
                    Coeffa0|
                    Coeffa1|
                    Coeffa2|
                    Coeffa3|
                    CoeffOfDetermination|
                    CoeffOfDeterminationLimitLow|
                    CoeffOfDeterminationLimitType|
                    Comment|
                    CorrelationCoeff|
                    CorrelationCoeffLimitLow|
                    CorrelationCoeffLimitType|
                    DifferenceErrorRatio|
                    Efficiency|
                    Inclusion|
                    LabQualifiers|
                    Mass|
                    MassLimitHigh|
                    MassLimitLow|
                    MassLimitType|
                    MassUnits|
                    MeanCalibrationFactor|
                    MeanCalibrationFactorUnits|
                    MeanRetentionTime|
                    MeanRetentionTimeLimitHigh|
                    MeanRetentionTimeLimitLow|
                    MeanRetentionTimeLimitType|
                    MeanRetentionTimeUnits|
```

```
                MeanRRF|
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                MeanRRFLimitType|
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                PercentDifferenceLimitLow|
                PercentDifferenceLimitType|
                PercentRecovery|
                PercentRecoveryLimitHigh|
                PercentRecoveryLimitLow|
                PercentRecoveryLimitType|
                PercentRecoveryType|
                PercentRSD|
                PercentRSDLimitHigh|
                PercentRSDLimitLow|
                PercentRSDLimitType|
                Resolution|
                ResolutionLimitHigh|
                ResolutionLimitLow|
                ResolutionLimitType|
                ResolutionType|
                ResolutionUnits|
                Result|
                ResultLimitHigh|
                ResultLimitLow|
                ResultLimitType|
                ResultType|
                ResultUncertainty|
                ResultUnits|
                RRF|
                RRFLimitLow|
                RRFLimitType|
                TailingFactor|
                TailingFactorLimitHigh|
                TailingFactorLimitType|
                Wavelength|
                WavelengthUnits|
                WeightingFactor|
                PeakComparison
                        )*>
<!ELEMENT PeakComparison (
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                PercentRatio|
                PercentRatioLimitHigh|
                PercentRatioLimitLow|
                PercentRatioLimitType
                        )*>
<!ELEMENT PreparationPlusCleanup (
                AliquotAmount|
                AliquotAmountUnits|
                Analyst|
                CleanedUpDate|
                CleanupBatch|
                CleanupType|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
```

```
                    ClientMethodSource|
                    ClientMethodVersion|
                    Comment|
                    FinalAmount|
                    FinalAmountUnits|
                    InitialAmount|
                    InitialAmountUnits|
                    LabID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    LotNumber|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    PreparationBatch|
                    PreparationPlusCleanupType|
                    PreparationType|
                    PreparedDate|
                    ProcedureID|
                    ProcedureName|
                    Solvent|
                    Characteristic
                              )*>
<!ELEMENT ReportedResult (
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                    AnalyteName|
                    AnalyteNameContext|
                    AnalyteType|
                    BiasErrorRatio|
                    CASRegistryNumber|
                    ClientAnalyteID|
                    ClientAnalyteName|
                    ClientDetectionLimit|
                    ClientDetectionLimitUnits|
                    ClientQuantitationLimit|
                    ClientQuantitationLimitUnits|
                    Comment|
                    DetectionLimit|
                    DetectionLimitType|
                    DetectionLimitUnits|
                    DifferenceErrorRatio|
                    ExpectedResult|
                    ExpectedResultUncertainty|
                    ExpectedResultUncertaintyConfidenceLevel|
                    ExpectedResultUncertaintyDetermination|
                    ExpectedResultUncertaintyIntervalType|
                    ExpectedResultUncertaintyLimitHigh|
                    ExpectedResultUncertaintyLimitLow|
                    ExpectedResultUncertaintyType
                    ExpectedResultUncertaintyUnits|
                    ExpectedResultUnits|
                    LabAnalysisID|
                    LabAnalyteID|
                    LabQualifiers|
                    LabResultStatus|
```

```
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                        PercentDifferenceLimitLow|
                        PercentDifferenceLimitType|
                        PercentRecovery|
                        PercentRecoveryLimitHigh|
                        PercentRecoveryLimitLow|
                        PercentRecoveryLimitType|
                        PercentRecoveryType|
                        QuantitationLimit|
                        QuantitationLimitType|
                        QuantitationLimitUnits|
                        ReportingLimit|
                        ReportingLimitType|
                        ReportingLimitUnits|
                        Result|
                        ResultLimitHigh|
                        ResultLimitLow|
                        ResultLimitType|
                        ResultType|
                        ResultUncertainty|
                        ResultUncertaintyConfidenceLevel|
                        ResultUncertaintyDetermination|
                        ResultUncertaintyIntervalType|
                        ResultUncertaintyLimitHigh|
                        ResultUncertaintyLimitLow|
                        ResultUncertaintyType|
                        ResultUncertaintyUnits|
                        ResultUnits|
                        RetentionTime|
                        RetentionTimeUnits|
                        RPD|
                        RPDLimitHigh|
                        RPDLimitType|
                        RPDType
                                )*>
<!ELEMENT SamplePlusMethod (
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                        ClientMethodCategory|
                        ClientMethodCode|
                        ClientMethodID|
                        ClientMethodModificationDescription|
                        ClientMethodModificationID|
                        ClientMethodName|
                        ClientMethodSource|
                        ClientMethodType|
                        ClientMethodVersion|
                        ClientName|
                        ClientSampleID|
                        CollectedDate|
                        CollectedEndDate|
                        Comment|
                        Composite|
                        CoolerID|
                        CustodyID|
                        EquipmentBatch|
                        Filtered|
                        LabContract|
                        LabContractModificationDescription|
                        LabContractModificationID|
```

```
                    LabID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    LabReceiptDate|
                    LabReportingBatch|
                    LabSampleID|
                    LocationID|
                    LocationName|
                    MatrixID|
                    MatrixMedium|
                    MethodBatch|
                    MethodCategory|
                    MethodCode|
                    MethodID|
                    MethodLevel|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodType|
                    MethodVersion|
                    OriginalClientSampleID|
                    OriginalLabSampleID|
                    PhaseAnalyzed|
                    Preservative|
                    ProjectID|
                    ProjectName|
                    QCCategory|
                    QCLinkage|
                    QCType|
                    Quarantine|
                    SamplingBatch|
                    ShippingBatch|
                    SiteID|
                    SiteName|
                    StorageBatch|
                    Analysis|
                    Characteristic|
                    ReportedResult|
                    Handling|
                    AnalysisGroup
                              )*>
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```

```
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<!ELEMENT PercentRatioLimitLow (#PCDATA)>
<!ELEMENT PercentRatioLimitType (#PCDATA)>
<!ELEMENT PercentRecovery (#PCDATA)>
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```

```
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<!ELEMENT Wavelength (#PCDATA)>
<!ELEMENT WavelengthUnits (#PCDATA)>
<!ELEMENT WeightingFactor (#PCDATA)>
<!ELEMENT Yield (#PCDATA)>
```

6.4   General Stage 2a DTD

```
<?xml version="1.0" encoding="UTF-8"?>
<!—SEDD_5-2_GENERAL_2a_2.dtd 07/21/2008 Based on SEDD Specification 5.2 -->
<!-- Acronym Description -->
<!-- EDD   - Electronic Data Deliverable -->
<!-- ID    - Identity -->
<!-- Lab   - Laboratory -->
<!-- QC    - Quality Control -->
<!-- RPD   - Relative Percent Difference -->
<!ELEMENT Header (
                ClientID|
                ClientName|
                Comment|
                DateFormat|
                EDDID|
                EDDImplementationID|
                EDDImplementationVersion|
                EDDVersion|
                GeneratingSystemID|
                GeneratingSystemVersion|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabDataPackageID|
                LabDataPackageName|
                LabDataPackageVersion|
                LabID|
                LabName|
                LabNarrative|
                LabQualifiersDefinition|
                LabReportedDate|
                ProjectID|
                ProjectName|
                SiteID|
                SiteName|
                ContactInformation|
                SamplePlusMethod
                        )*>
<!ELEMENT Analysis (
                AliquotAmount|
                AliquotAmountUnits|
                AnalysisDuration|
                AnalysisDurationUnits|
                AnalysisGroupID|
                AnalysisType|
                Analyst|
                AnalyzedAmount|
                AnalyzedAmountUnits|
                AnalyzedDate|
                ClientAnalysisID|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodVersion|
                Column|
                ColumnInternalDiameter|
```

```
                        ColumnInternalDiameterUnits|
                        ColumnLength|
                        ColumnLengthUnits|
                        Comment|
                        ConfirmationAnalysisID|
                        Counts|
                        CountsUncertainty|
                        CountsUncertaintyConfidenceLevel|
                        CountsUncertaintyDetermination|
                        CountsUncertaintyIntervalType|
                        CountsUncertaintyLimitHigh|
                        CountsUncertaintyLimitLow|
                        CountsUncertaintyType|
                        CountsUnits|
                        DetectorID|
                        DetectorType|
                        DilutionFactor|
                        Efficiency|
                        HeatedPurge|
                        Inclusion|
                        InjectionVolume|
                        InjectionVolumeUnits|
                        InstrumentID|
                        LabAnalysisID|
                        LabFileID|
                        LabID|
                        LabMethodID|
                        LabMethodName|
                        LabName|
                        MethodCode|
                        MethodID|
                        MethodModificationDescription|
                        MethodModificationID|
                        MethodName|
                        MethodSource|
                        MethodVersion|
                        PreparationBatch|
                        ProcedureID|
                        ProcedureName|
                        ReferenceDate|
                        ResultBasis|
                        Temperature|
                        TemperatureUnits|
                        Wavelength|
                        WavelengthUnits|
                        Yield|
                        PreparationPlusCleanup|
                        Analyte|
                        AnalyteGroup
                                )*>
<!ELEMENT AnalysisGroup (
                        AnalysisGroupID|
                        AnalysisType|
                        Comment|
                        Analyte|
                        AnalyteGroup
                                )*>
```

```
<!ELEMENT Analyte (
              AnalyteGroupID|
              AnalyteName|
              AnalyteNameContext|
              AnalyteType|
              CASRegistryNumber|
              ClientAnalyteID|
              ClientAnalyteName|
              Comment|
              Counts|
              CountsUncertainty|
              CountsUncertaintyConfidenceLevel|
              CountsUncertaintyDetermination|
              CountsUncertaintyIntervalType|
              CountsUncertaintyLimitHigh|
              CountsUncertaintyLimitLow|
              CountsUncertaintyType|
              CountsUnits|
              DetectionLimit|
              DetectionLimitType|
              DetectionLimitUnits|
              DifferenceErrorRatio|
              Efficiency|
              ExpectedResult|
              ExpectedResultUncertainty|
              ExpectedResultUncertaintyConfidenceLevel|
              ExpectedResultUncertaintyDetermination|
              ExpectedResultUncertaintyIntervalType|
              ExpectedResultUncertaintyLimitHigh|
              ExpectedResultUncertaintyLimitLow|
              ExpectedResultUncertaintyType|
              ExpectedResultUncertaintyUnits|
              ExpectedResultUnits|
              Inclusion|
              LabAnalyteID|
              LabQualifiers|
              LotNumber|
              PeakID|
              PercentRecovery|
              PercentRecoveryLimitHigh|
              PercentRecoveryLimitLow|
              PercentRecoveryLimitType|
              PercentRecoveryType|
              QuantitationLimit|
              QuantitationLimitType|
              QuantitationLimitUnits|
              ReportingLimit|
              ReportingLimitType|
              ReportingLimitUnits|
              Result|
              ResultLimitHigh|
              ResultLimitLow|
              ResultLimitType|
              ResultType|
              ResultUncertainty|
              ResultUncertaintyConfidenceLevel|
              ResultUncertaintyDetermination|
              ResultUncertaintyIntervalType|
              ResultUncertaintyLimitHigh|
              ResultUncertaintyLimitLow|
              ResultUncertaintyType|
```

```
                        ResultUncertaintyUnits|
                        ResultUnits|
                        StandardSource|
                        Wavelength|
                        WavelengthUnits
                                )*>
<!ELEMENT AnalyteGroup (
                        AnalyteGroupID|
                        AnalyteName|
                        AnalyteNameContext|
                        AnalyteType|
                        CASRegistryNumber|
                        ClientAnalyteID|
                        ClientAnalyteName|
                        Comment|
                        LabAnalyteID|
                        LabQualifiers|
                        Result|
                        ResultType|
                        ResultUncertainty|
                        ResultUnits
                                )*>
<!ELEMENT Characteristic (
                        CharacteristicType|
                        CharacteristicValue|
                        CharacteristicUnits|
                        Comment
                                )*>
<!ELEMENT ContactInformation (
                        LabAddress1|
                        LabAddress2|
                        LabCity|
                        LabCountry|
                        LabID|
                        LabName|
                        LabPointOfContact|
                        LabPointOfContactElectronicAddress|
                        LabPointOfContactTitle|
                        LabPointOfContactType|
                        LabState|
                        LabTelephoneNumber|
                        LabType|
                        LabZipCode
                                )*>
<!ELEMENT Handling (
                        Analyst|
                        ClientMethodCode|
                        ClientMethodID|
                        ClientMethodModificationDescription|
                        ClientMethodModificationID|
                        ClientMethodName|
                        ClientMethodSource|
                        ClientMethodVersion|
                        Comment|
                        HandledDate|
                        HandlingBatch|
                        HandlingType|
                        InitialAmount|
                        InitialAmountUnits|
                        LabID|
```

```
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    ProcedureID|
                    ProcedureName|
                    SampleAmount|
                    SampleAmountUnits|
                    Characteristic
                            )*>
<!ELEMENT PreparationPlusCleanup (
                    AliquotAmount|
                    AliquotAmountUnits|
                    Analyst|
                    CleanedUpDate|
                    CleanupBatch|
                    CleanupType|
                    ClientMethodCode|
                    ClientMethodID|
                    ClientMethodModificationDescription|
                    ClientMethodModificationID|
                    ClientMethodName|
                    ClientMethodSource|
                    ClientMethodVersion|
                    Comment|
                    FinalAmount|
                    FinalAmountUnits|
                    InitialAmount|
                    InitialAmountUnits|
                    LabID|
                    LabMethodID|
                    LabMethodName|
                    LabName|
                    LotNumber|
                    MethodCode|
                    MethodID|
                    MethodModificationDescription|
                    MethodModificationID|
                    MethodName|
                    MethodSource|
                    MethodVersion|
                    PreparationBatch|
                    PreparationPlusCleanupType|
                    PreparationType|
                    PreparedDate|
                    ProcedureID|
                    ProcedureName|
                    Solvent|
                    Characteristic
                            )*>
<!ELEMENT ReportedResult (
                    AnalysisGroupID|
                    AnalyteGroupID|
                    AnalyteName|
```

```
AnalyteNameContext|
AnalyteType|
BiasErrorRatio|
CASRegistryNumber|
ClientAnalyteID|
ClientAnalyteName|
ClientDetectionLimit|
ClientDetectionLimitUnits|
ClientQuantitationLimit|
ClientQuantitationLimitUnits|
Comment|
DetectionLimit|
DetectionLimitType|
DetectionLimitUnits|
DifferenceErrorRatio|
ExpectedResult|
ExpectedResultUncertainty|
ExpectedResultUncertaintyConfidenceLevel|
ExpectedResultUncertaintyDetermination|
ExpectedResultUncertaintyIntervalType|
ExpectedResultUncertaintyLimitHigh|
ExpectedResultUncertaintyLimitLow|
ExpectedResultUncertaintyType|
ExpectedResultUncertaintyUnits|
ExpectedResultUnits|
LabAnalysisID|
LabAnalyteID|
LabQualifiers|
LabResultStatus|
PeakID|
PercentDifference|
PercentDifferenceLimitHigh|
PercentDifferenceLimitLow|
PercentDifferenceLimitType|
PercentRecovery|
PercentRecoveryLimitHigh|
PercentRecoveryLimitLow|
PercentRecoveryLimitType|
PercentRecoveryType|
QuantitationLimit|
QuantitationLimitType|
QuantitationLimitUnits|
ReportingLimit|
ReportingLimitType|
ReportingLimitUnits|
Result|
ResultLimitHigh|
ResultLimitLow|
ResultLimitType|
ResultType|
ResultUncertainty|
ResultUncertaintyConfidenceLevel|
ResultUncertaintyDetermination|
ResultUncertaintyIntervalType|
ResultUncertaintyLimitHigh|
ResultUncertaintyLimitLow|
ResultUncertaintyType|
ResultUncertaintyUnits|
```

```
                ResultUnits|
                RetentionTime|
                RetentionTimeUnits|
                RPD|
                RPDLimitHigh|
                RPDLimitType|
                RPDType
                        )*>
<!ELEMENT SamplePlusMethod (
                ClientID|
                ClientMethodCategory|
                ClientMethodCode|
                ClientMethodID|
                ClientMethodModificationDescription|
                ClientMethodModificationID|
                ClientMethodName|
                ClientMethodSource|
                ClientMethodType|
                ClientMethodVersion|
                ClientName|
                ClientSampleID|
                CollectedDate|
                CollectedEndDate|
                Comment|
                Composite|
                CoolerID|
                CustodyID|
                EquipmentBatch|
                Filtered|
                LabContract|
                LabContractModificationDescription|
                LabContractModificationID|
                LabID|
                LabMethodID|
                LabMethodName|
                LabName|
                LabReceiptDate|
                LabReportingBatch|
                LabSampleID|
                LocationID|
                LocationName|
                MatrixID|
                MatrixMedium|
                MethodBatch|
                MethodCategory|
                MethodCode|
                MethodID|
                MethodLevel|
                MethodModificationDescription|
                MethodModificationID|
                MethodName|
                MethodSource|
                MethodType|
                MethodVersion|
                OriginalClientSampleID|
                OriginalLabSampleID|
                PhaseAnalyzed|
                Preservative|
                ProjectID|
```

```
                      ProjectName|
                      QCCategory|
                      QCLinkage|
                      QCType|
                      Quarantine|
                      SamplingBatch|
                      ShippingBatch|
                      SiteID|
                      SiteName|
                      StorageBatch|
                      Analysis|
                      Characteristic|
                      ReportedResult|
                      Handling|
                      AnalysisGroup
                                )*>
<!ELEMENT AliquotAmount (#PCDATA)>
<!ELEMENT AliquotAmountUnits (#PCDATA)>
<!ELEMENT AnalysisDuration (#PCDATA)>
<!ELEMENT AnalysisDurationUnits (#PCDATA)>
<!ELEMENT AnalysisGroupID (#PCDATA)>
<!ELEMENT AnalysisType (#PCDATA)>
<!ELEMENT Analyst (#PCDATA)>
<!ELEMENT AnalyteGroupID (#PCDATA)>
<!ELEMENT AnalyteName (#PCDATA)>
<!ELEMENT AnalyteNameContext (#PCDATA)>
<!ELEMENT AnalyteType (#PCDATA)>
<!ELEMENT AnalyzedAmount (#PCDATA)>
<!ELEMENT AnalyzedAmountUnits (#PCDATA)>
<!ELEMENT AnalyzedDate (#PCDATA)>
<!ELEMENT BiasErrorRatio (#PCDATA)>
<!ELEMENT CASRegistryNumber (#PCDATA)>
<!ELEMENT CharacteristicType (#PCDATA)>
<!ELEMENT CharacteristicUnits (#PCDATA)>
<!ELEMENT CharacteristicValue (#PCDATA)>
<!ELEMENT CleanedUpDate (#PCDATA)>
<!ELEMENT CleanupBatch (#PCDATA)>
<!ELEMENT CleanupType (#PCDATA)>
<!ELEMENT ClientAnalysisID (#PCDATA)>
<!ELEMENT ClientAnalyteID (#PCDATA)>
<!ELEMENT ClientAnalyteName (#PCDATA)>
<!ELEMENT ClientDetectionLimit (#PCDATA)>
<!ELEMENT ClientDetectionLimitUnits (#PCDATA)>
<!ELEMENT ClientID (#PCDATA)>
<!ELEMENT ClientMethodCategory (#PCDATA)>
<!ELEMENT ClientMethodCode (#PCDATA)>
<!ELEMENT ClientMethodID (#PCDATA)>
<!ELEMENT ClientMethodModificationDescription (#PCDATA)>
<!ELEMENT ClientMethodModificationID (#PCDATA)>
<!ELEMENT ClientMethodName (#PCDATA)>
<!ELEMENT ClientMethodSource (#PCDATA)>
<!ELEMENT ClientMethodType (#PCDATA)>
<!ELEMENT ClientMethodVersion (#PCDATA)>
<!ELEMENT ClientName (#PCDATA)>
<!ELEMENT ClientQuantitationLimit (#PCDATA)>
<!ELEMENT ClientQuantitationLimitUnits (#PCDATA)>
<!ELEMENT ClientSampleID (#PCDATA)>
<!ELEMENT CollectedDate (#PCDATA)>
<!ELEMENT CollectedEndDate (#PCDATA)>
```

```
<!ELEMENT Column (#PCDATA)>
<!ELEMENT ColumnInternalDiameter (#PCDATA)>
<!ELEMENT ColumnInternalDiameterUnits (#PCDATA)>
<!ELEMENT ColumnLength (#PCDATA)>
<!ELEMENT ColumnLengthUnits (#PCDATA)>
<!ELEMENT Comment (#PCDATA)>
<!ELEMENT Composite (#PCDATA)>
<!ELEMENT ConfirmationAnalysisID (#PCDATA)>
<!ELEMENT CoolerID (#PCDATA)>
<!ELEMENT Counts (#PCDATA)>
<!ELEMENT CountsUncertainty (#PCDATA)>
<!ELEMENT CountsUncertaintyConfidenceLevel (#PCDATA)>
<!ELEMENT CountsUncertaintyDetermination (#PCDATA)>
<!ELEMENT CountsUncertaintyIntervalType (#PCDATA)>
<!ELEMENT CountsUncertaintyLimitHigh (#PCDATA)>
<!ELEMENT CountsUncertaintyLimitLow (#PCDATA)>
<!ELEMENT CountsUncertaintyType (#PCDATA)>
<!ELEMENT CountsUnits (#PCDATA)>
<!ELEMENT CustodyID (#PCDATA)>
<!ELEMENT DateFormat (#PCDATA)>
<!ELEMENT DetectionLimit (#PCDATA)>
<!ELEMENT DetectionLimitType (#PCDATA)>
<!ELEMENT DetectionLimitUnits (#PCDATA)>
<!ELEMENT DetectorID (#PCDATA)>
<!ELEMENT DetectorType (#PCDATA)>
<!ELEMENT DifferenceErrorRatio (#PCDATA)>
<!ELEMENT DilutionFactor (#PCDATA)>
<!ELEMENT EDDID (#PCDATA)>
<!ELEMENT EDDImplementationID (#PCDATA)>
<!ELEMENT EDDImplementationVersion (#PCDATA)>
<!ELEMENT EDDVersion (#PCDATA)>
<!ELEMENT Efficiency (#PCDATA)>
<!ELEMENT EquipmentBatch (#PCDATA)>
<!ELEMENT ExpectedResult (#PCDATA)>
<!ELEMENT ExpectedResultUncertainty (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyConfidenceLevel (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyDetermination (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyIntervalType (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyLimitHigh (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyLimitLow (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyType (#PCDATA)>
<!ELEMENT ExpectedResultUncertaintyUnits (#PCDATA)>
<!ELEMENT ExpectedResultUnits (#PCDATA)>
<!ELEMENT Filtered (#PCDATA)>
<!ELEMENT FinalAmount (#PCDATA)>
<!ELEMENT FinalAmountUnits (#PCDATA)>
<!ELEMENT GeneratingSystemID (#PCDATA)>
<!ELEMENT GeneratingSystemVersion (#PCDATA)>
<!ELEMENT HandledDate (#PCDATA)>
<!ELEMENT HandlingBatch (#PCDATA)>
<!ELEMENT HandlingType (#PCDATA)>
<!ELEMENT HeatedPurge (#PCDATA)>
<!ELEMENT Inclusion (#PCDATA)>
<!ELEMENT InitialAmount (#PCDATA)>
<!ELEMENT InitialAmountUnits (#PCDATA)>
<!ELEMENT InjectionVolume (#PCDATA)>
<!ELEMENT InjectionVolumeUnits (#PCDATA)>
<!ELEMENT InstrumentID (#PCDATA)>
<!ELEMENT LabAddress1 (#PCDATA)>
```

```
<!ELEMENT LabAddress2 (#PCDATA)>
<!ELEMENT LabAnalysisID (#PCDATA)>
<!ELEMENT LabAnalyteID (#PCDATA)>
<!ELEMENT LabCity (#PCDATA)>
<!ELEMENT LabContract (#PCDATA)>
<!ELEMENT LabContractModificationDescription (#PCDATA)>
<!ELEMENT LabContractModificationID (#PCDATA)>
<!ELEMENT LabCountry (#PCDATA)>
<!ELEMENT LabDataPackageID (#PCDATA)>
<!ELEMENT LabDataPackageName (#PCDATA)>
<!ELEMENT LabDataPackageVersion (#PCDATA)>
<!ELEMENT LabFileID (#PCDATA)>
<!ELEMENT LabID (#PCDATA)>
<!ELEMENT LabMethodID (#PCDATA)>
<!ELEMENT LabMethodName (#PCDATA)>
<!ELEMENT LabName (#PCDATA)>
<!ELEMENT LabNarrative (#PCDATA)>
<!ELEMENT LabPointOfContact (#PCDATA)>
<!ELEMENT LabPointOfContactElectronicAddress (#PCDATA)>
<!ELEMENT LabPointOfContactTitle (#PCDATA)>
<!ELEMENT LabPointOfContactType (#PCDATA)>
<!ELEMENT LabQualifiers (#PCDATA)>
<!ELEMENT LabQualifiersDefinition (#PCDATA)>
<!ELEMENT LabReceiptDate (#PCDATA)>
<!ELEMENT LabReportedDate (#PCDATA)>
<!ELEMENT LabReportingBatch (#PCDATA)>
<!ELEMENT LabResultStatus (#PCDATA)>
<!ELEMENT LabSampleID (#PCDATA)>
<!ELEMENT LabState (#PCDATA)>
<!ELEMENT LabTelephoneNumber (#PCDATA)>
<!ELEMENT LabType (#PCDATA)>
<!ELEMENT LabZipCode (#PCDATA)>
<!ELEMENT LocationID (#PCDATA)>
<!ELEMENT LocationName (#PCDATA)>
<!ELEMENT LotNumber (#PCDATA)>
<!ELEMENT MatrixID (#PCDATA)>
<!ELEMENT MatrixMedium (#PCDATA)>
<!ELEMENT MethodBatch (#PCDATA)>
<!ELEMENT MethodCategory (#PCDATA)>
<!ELEMENT MethodCode (#PCDATA)>
<!ELEMENT MethodID (#PCDATA)>
<!ELEMENT MethodLevel (#PCDATA)>
<!ELEMENT MethodModificationDescription (#PCDATA)>
<!ELEMENT MethodModificationID (#PCDATA)>
<!ELEMENT MethodName (#PCDATA)>
<!ELEMENT MethodSource (#PCDATA)>
<!ELEMENT MethodType (#PCDATA)>
<!ELEMENT MethodVersion (#PCDATA)>
<!ELEMENT OriginalClientSampleID (#PCDATA)>
<!ELEMENT OriginalLabSampleID (#PCDATA)>
<!ELEMENT PeakID (#PCDATA)>
<!ELEMENT PercentDifference (#PCDATA)>
<!ELEMENT PercentDifferenceLimitHigh (#PCDATA)>
<!ELEMENT PercentDifferenceLimitLow (#PCDATA)>
<!ELEMENT PercentDifferenceLimitType (#PCDATA)>
<!ELEMENT PercentRecovery (#PCDATA)>
<!ELEMENT PercentRecoveryLimitHigh (#PCDATA)>
<!ELEMENT PercentRecoveryLimitLow (#PCDATA)>
<!ELEMENT PercentRecoveryLimitType (#PCDATA)>
```

```
<!ELEMENT PercentRecoveryType (#PCDATA)>
<!ELEMENT PhaseAnalyzed (#PCDATA)>
<!ELEMENT PreparationBatch (#PCDATA)>
<!ELEMENT PreparationPlusCleanupType (#PCDATA)>
<!ELEMENT PreparationType (#PCDATA)>
<!ELEMENT PreparedDate (#PCDATA)>
<!ELEMENT Preservative (#PCDATA)>
<!ELEMENT ProcedureID (#PCDATA)>
<!ELEMENT ProcedureName (#PCDATA)>
<!ELEMENT ProjectID (#PCDATA)>
<!ELEMENT ProjectName (#PCDATA)>
<!ELEMENT QCCategory (#PCDATA)>
<!ELEMENT QCLinkage (#PCDATA)>
<!ELEMENT QCType (#PCDATA)>
<!ELEMENT QuantitationLimit (#PCDATA)>
<!ELEMENT QuantitationLimitType (#PCDATA)>
<!ELEMENT QuantitationLimitUnits (#PCDATA)>
<!ELEMENT Quarantine (#PCDATA)>
<!ELEMENT ReferenceDate (#PCDATA)>
<!ELEMENT ReportingLimit (#PCDATA)>
<!ELEMENT ReportingLimitType (#PCDATA)>
<!ELEMENT ReportingLimitUnits (#PCDATA)>
<!ELEMENT Result (#PCDATA)>
<!ELEMENT ResultBasis (#PCDATA)>
<!ELEMENT ResultLimitHigh (#PCDATA)>
<!ELEMENT ResultLimitLow (#PCDATA)>
<!ELEMENT ResultLimitType (#PCDATA)>
<!ELEMENT ResultType (#PCDATA)>
<!ELEMENT ResultUncertainty (#PCDATA)>
<!ELEMENT ResultUncertaintyConfidenceLevel (#PCDATA)>
<!ELEMENT ResultUncertaintyDetermination (#PCDATA)>
<!ELEMENT ResultUncertaintyIntervalType (#PCDATA)>
<!ELEMENT ResultUncertaintyLimitHigh (#PCDATA)>
<!ELEMENT ResultUncertaintyLimitLow (#PCDATA)>
<!ELEMENT ResultUncertaintyType (#PCDATA)>
<!ELEMENT ResultUncertaintyUnits (#PCDATA)>
<!ELEMENT ResultUnits (#PCDATA)>
<!ELEMENT RetentionTime (#PCDATA)>
<!ELEMENT RetentionTimeUnits (#PCDATA)>
<!ELEMENT RPD (#PCDATA)>
<!ELEMENT RPDLimitHigh (#PCDATA)>
<!ELEMENT RPDLimitType (#PCDATA)>
<!ELEMENT RPDType (#PCDATA)>
<!ELEMENT SampleAmount (#PCDATA)>
<!ELEMENT SampleAmountUnits (#PCDATA)>
<!ELEMENT SamplingBatch (#PCDATA)>
<!ELEMENT ShippingBatch (#PCDATA)>
<!ELEMENT SiteID (#PCDATA)>
<!ELEMENT SiteName (#PCDATA)>
<!ELEMENT Solvent (#PCDATA)>
<!ELEMENT StandardSource (#PCDATA)>
<!ELEMENT StorageBatch (#PCDATA)>
<!ELEMENT Temperature (#PCDATA)>
<!ELEMENT TemperatureUnits (#PCDATA)>
<!ELEMENT Wavelength (#PCDATA)>
<!ELEMENT WavelengthUnits (#PCDATA)>
<!ELEMENT Yield (#PCDATA)>
```

7.0   DATA ELEMENT INSTRUCTION TABLES

Column abbreviations: Matrix Spike (MS), Duplicate Sample (Dup), Laboratory Control Sample (LCS), Preparation Blank (PB), Leachate Extraction Blank (LEB), Post-Digestion/Distillation Spike (PDS), Serial Dilution (SD), Non-Client Sample (NCS), Instrument Performance Check (IPC), Initial Calibration (ICAL), Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Interference Check Sample (ICS).

7.1   Stage 3

TABLE 1. INORGANICS DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
Header	X	X	X	X	X	X	X	X	
ClientID	X	X	X	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName									Not required.
Comment									Not required.
DateFormat	X	X	X	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabDataPackageID	X	X	X	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabNarrative									Not required.
LabQualifiersDefinition	X	X	X	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	X	X	X	Report the date this data was reported to the client.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
ProjectID	X	X	X	X	X	X	X	X	Report the Case Number.
ProjectName									Not required.
SiteID									Not required.
SiteName									Not required.
SamplePlusMethod	X	X	X	X	X	X	X	X	
Bottles									Not required.
BottleType									Not required.
ClientID	X	X	X						Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory									Not required.
ClientMethodCode	X	X	X	X	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID	X	X	X	X	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
ClientMethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName									Not required.
ClientSampleID	X	X	X	X	X	X	X	X	Report the EPA Sample Number.
CollectedDate	X	X	X						Report the date and time the sample was collected.
CollectedEndDate									Not required.
Comment									Not required.
Composite									Not required.
CoolerID									Not required.
CustodyID	X								Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch									Not required.
Filtered	X								Report "Yes" for dissolved metals, or "No" for total metals.
LabContract	X	X	X	X	X	X	X		Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID									Not required.
LabMethodName									Not required.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
LabReceiptDate	X	X	X						Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID									Not required.
LocationName									Not required.
MatrixID	X	X	X	X	X	X	X	X	Report "Water", "Soil", or "Wipe" as applicable.
MatrixMedium	X	X	X	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable. Use "Solid" for wipes.
MethodBatch									Not required.
MethodCategory									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
MethodLevel									Not required.
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X	X			X	X		Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID									Not required.
PhaseAnalyzed									Not required.
Preservative	X	X	X						Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X	X	X	X		Report the Case Number.
ProjectName									Not required.
QCCategory		X	X	X	X	X	X		Report "Blank" for PB and LEB, "Spike" for MS and post-digestion spike, "Blank_Spike" for LCS, "Duplicate" for duplicate, or "Serial_Dilution" for SD.
QCLinkage		X	X	X	X	X	X		Report "LabReportingBatch" for MS, post-digestion spike, Dup, and SD; "PreparationBatch" for PB and LCS or "HandlingBatch" for LEB.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
									Applicability
QCType	X	X	X	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "PT_Sample" for Performance Evaluation samples or Proficiency Testing audit samples; "Method_Blank" for PB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Duplicate" for Dup; "Laboratory_Control_Sample" for LCS; "Post_Digestion_Spike" for post-digestion spikes; "Serial_Dilution" for SD; or "Non_Client_Sample" for NCS.
Quarantine	X								Report "Yes" or "No" based on sampling information.
SamplingBatch									Not required.
ShippingBatch									Not required.
SiteID									Not required.
SiteName									Not required.
StorageBatch									Not required.
InstrumentQC									Not required.
Characteristic	X	X	X	X	X	X	X		
CharacteristicType	X	X	X	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Report the "pH" and "Temperature" measured for the TCLP or SPLP leachates under the Handling node. Tissue samples do not require "Percent_Solids" or "pH". Wipe samples do not require "Percent_Solids", "pH", or "Temperature".
CharacteristicValue	X	X	X	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X	X	X	X		Report "C" for "Temperature".
Comment									Not required.
ContactInformation	X	X	X	X	X	X	X	X	
LabAddress1	X	X	X	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
LabCity	X	X	X	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType									Not required.
LabState	X	X	X	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType									Not required.
LabZipCode	X	X	X	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	X	X	X	
AliquotAmount									Not required.
AliquotAmountUnits									Not required.
AnalysisBatch	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration									Not required.
AnalysisDurationUnits									Not required.
AnalysisGroupID									Not required.
AnalysisType	X	X	X	X	X	X	X		Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.
Analyst	X	X	X	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount									Not required.
AnalyzedAmountUnits									Not required.
AnalyzedDate	X	X	X	X	X	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection	X	X	X	X	X	X	X		For ICP-AES and ICP-MS, enter "Yes" if background correction were applied; otherwise enter "No".
BackgroundRawData	X	X	X	X	X	X	X		For ICP-AES and ICP-MS, enter "Yes" if background corrections applied before raw data generated. Otherwise enter "No".
BackgroundType									Not required.

| Node and Data Elements | Applicability | | | | | | | | Instructions |
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
BottleID									Not required.
ClientAnalysisID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
Column									Not required.
ColumnInternalDiameter									Not required.
ColumnInternalDiameterUnits									Not required.
ColumnLength									Not required.
ColumnLengthUnits									Not required.
Comment									Not required.
ConfirmationAnalysisID									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectorID									Not required.
DetectorType									Not required.
DilutionFactor	X	X	X	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency									Not required.
HeatedPurge									Not required.
Inclusion									Not required.
InjectionVolume									Not required.
InjectionVolumeUnits									Not required.
InstrumentID	X	X	X	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection	X	X	X	X	X	X	X		For ICP-AES and ICP-MS, enter "Yes" if interelement corrections were applied; otherwise enter "No".
LabAnalysisID	X	X	X	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	X	X	X	Report the lab file ID.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalLabAnalysisID	X	X	X			X	X		If a dilution or reanalysis of a previously analyzed sample is performed, report the Lab Analysis ID of the original sample that was used for the dilution or reanalysis.
PreparationBatch									Not required.
ProcedureID									Not required.
ProcedureName									Not required.
ReferenceDate									Not required.
ResultBasis	X	X	X	X	X				Report "Dry" for soil/sediment samples. For aqueous/water samples, report "Dissolved" if sample field-filtered; otherwise report "Total". Report "Wet" for tissue samples or for any other matrices (not aqueous/water) for which the results are not corrected for percent solids.
RunBatch	X	X	X	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL (Initial Calibration) sequence.
SampleAmount									Not required.
SampleAmountUnits									Not required.
Temperature									Not required.
TemperatureUnits									Not required.
Wavelength									Not required.
WavelengthUnits									Not required.
Yield									Not required.
AnalysisGroup									Not required.
Handling	X	X	X		X				
Analyst									Not required.
BottleID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X		X				Report "ISM02.4".

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X		X				Report "EPA_CLP".
ClientMethodVersion	X	X	X		X				Report the month and year the SOW was issued.
Comment									Not required.
HandledDate	X	X	X		X				Enter the date and time TCLP or SPLP extraction began.
HandlingBatch	X	X	X		X				Links all samples that were TCLP or SPLP extracted together. Report a unique identifier for each batch.
HandlingType	X	X	X		X				Report "TCLP" or "SPLP".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X		X				Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X		X				Report "EPA_CLP".
MethodVersion	X	X	X		X				Report month and year the SOW was issued.
ProcedureID									Not required.
ProcedureName									Not required.
SampleAmount									Not required.
SampleAmountUnits									Not required.
ReportedResult	X	X	X	X	X	X	X		
AnalysisGroupID									Not required.
AnalyteGroupID	X	X	X	X	X	X	X		Report the unique identifier from the AnalyteGroup the Hardness result is derived from.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS" (Chemical Abstract Service).
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness or "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS analyses. Report "Derived" for Hardness.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit									Not required.
ClientDetectionLimitUnits									Not required.
ClientQuantitationLimit	X	X	X	X	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
Comment									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures. Not required for Hardness.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.
ExpectedResult		X		X		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes or the true value for LCS.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits		X		X		X			Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water (or "mg/L" for TCLP), or "ug" for wipe samples.
LabAnalysisID	X	X	X	X	X	X	X		Report the unique identifier from the analysis this reported result was derived from. Not required for Hardness.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report flags as specified in the SOW. Include the Q qualifiers from Form 1-IN.
LabResultStatus	X	X	X						Report "Preliminary" or "Final" as applicable.
PeakID									Not required.
PercentDifference							X		Report the Percent Difference to the nearest whole percent.
PercentDifferenceLimitHigh							X		Report the upper limit for the Percent Difference to the nearest whole percent.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
									Applicability
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType							X		Report "Method".
PercentRecovery		X		X		X			Report the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh		X		X					Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow		X		X					Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X		X					Report "Method".
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
RetentionTime									Not required.
RetentionTimeUnits									Not required.
RPD			X						Report the RPD to the nearest whole percent.
RPDLimitHigh			X						Report the upper limit for the RPD to the nearest whole percent.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
				Applicability					
RPDLimitType			X						Report "Method".
RPDType									Not required.
PreparationPlusCleanup	X	X	X	X	X	X	X		
AliquotAmount	X	X	X	X	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water to at least three significant figures. Not required for wipes.
AliquotAmountUnits	X	X	X	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water. Not required for wipes.
Analyst	X	X	X	X	X	X	X		Report the Analyst's initials.
BottleID									Not required.
CleanedUpDate									Not required.
CleanUpBatch									Not required.
CleanUpType									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X		Report the month and year the SOW was issued.
Comment									Not required.
Efficiency									Not required.
FinalAmount	X	X	X	X	X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits	X	X	X	X	X	X	X		Report "mL".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
LotNumber									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X		Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X		Report month and year the SOW was issued.

Node and Data Elements	Applicability							Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
PreparationBatch	X	X	X	X	X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType	X	X	X	X	X	X	X		Report "Preparation".
PreparationType	X	X	X	X	X	X	X		Report "Automated" or "Manual".
PreparedDate	X	X	X	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID									Not required.
ProcedureName									Not required.
SampleAmount									Not required.
SampleAmountUnits									Not required.
Solvent									Not required.
Analyte	X	X	X	X	X	X	X		
AmountAdded		X		X		X			Volume of spiking solution added in uL.
AmountAddedUnits		X		X		X			Report "uL".
AmountAddedLocation		X		X		X			For matrix spike report "Aliquot"; for LCS report "Standard"; for PDS report "Extracted_Aliquot".
AnalyteGroupID	X	X	X	X	X	X	X		Report the identifier that links the Ca or Mg result to the AnalyteGroup Hardness result.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness; "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS; "Internal_Standard" for internal standards; or "Monitor" for non-target interferences and masses requiring monitoring.
BiasErrorRatio									Not required.
CalibrationBasis									Not required.
CalibrationFactor									Not required.
CalibrationFactorUnits									Not required.
CalibrationType									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
Coeffa0									Not required.
Coeffa1									Not required.
Coeffa2									Not required.
Coeffa3									Not required.
CoeffOfDetermination									Not required.
CoeffOfDeterminationLimitLow									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
CoeffOfDeterminationLimitType									Not required.
Comment									Not required.
CorrelationCoeff									Not required.
CorrelationCoeffLimitLow									Not required.
CorrelationCoeffLimitType									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.
Efficiency									Not required.
ExpectedResult									Not required.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits									Not required.
Inclusion									Not required.
IntermediateResult	X	X	X	X	X	X	X		Report the raw concentration output of the instrument unadjusted for sample weight/volume, percent solids, or dilution factor.
IntermediateResultLimitHigh									Not required.
IntermediateResultLimitLow									Not required.
IntermediateResultLimitType									Not required.
IntermediateResultUnits	X	X	X	X	X	X	X		Report "ug/L".
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
LotNumber	X	X	X	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard (Internal Standards and spiking analytes only).
Mass									Not required.
MassLimitHigh									Not required.
MassLimitLow									Not required.
MassLimitType									Not required.
MassUnits									Not required.
MeanCalibrationFactor									Not required.
MeanCalibrationFactorUnits									Not required.
MeanRRF									Not required.
MeanRRFLimitLow									Not required.
MeanRRFLimitType									Not required.
PeakID	X	X	X	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown									Not required.
PercentBreakdownLimitHigh									Not required.
PercentBreakdownLimitType									Not required.
PercentDifference									Not required.
PercentDifferenceLimitHigh									Not required.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType									Not required.
PercentMatch									Not required.
PercentRecovery									Not required.
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
PercentRSD									Not required.
PercentRSDLimitHigh									Not required.
PercentRSDLimitLow									Not required.
PercentRSDLimitType									Not required.
QuantitationBasis									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Response									Not required.
ResponseLimitHigh									Not required.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
ResponseLimitLow									Not required.
ResponseLimitType									Not required.
ResponseUnits									Not required.
Result	X	X	X	X	X	X	X		For target or spike analyte detects, and for monitored masses, report the final calculated result.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, and "ug" for wipe samples.
RPD									Not required.
RPDLimitHigh									Not required.
RPDLimitType									Not required.
RPDType									Not required.
RRF									Not required.
RRFLimitLow									Not required.
RRFLimitType									Not required.
StandardConcentration	X	X	X	X	X	X	X		Report the concentration in ug/L of the standard added.
StandardConcentrationUnits	X	X	X	X	X	X	X		Report "ug/L".
StandardDeviation									Not required.
StandardDeviationUnits									Not required.
StandardFinalAmount									Not required.
StandardFinalAmountUnits									Not required.
StandardID									Not required.
StandardSource	X	X	X	X	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor									Not required.
TailingFactorLimitHigh									Not required.
TailingFactorLimitType									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
Wavelength									Not required.
WavelengthUnits									Not required.
WeightingFactor									Not required.
AnalyteComparison									Not required
AnalyteGroup	X	X	X	X	X	X	X		
AnalyteGroupID	X	X	X	X	X	X	X		Report a unique identifier.
AnalyteName	X	X	X	X	X	X	X		Report "Hardness".
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Derived".
CASRegistryNumber	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteID	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteName	X	X	X	X	X	X	X		Report "Hardness".
Comment									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW. Include the Q qualifiers from Form 1-IN.
Result	X	X	X	X	X	X	X		Report the final calculated for detects per the SOW.
ResultType	X	X	X	X	X	X	X		Report "=" for detects. Report "Not_Detected" for non-detects (where both Ca and Mg are not detected).
ResultUncertainty									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/L".
Peak	X	X	X	X	X	X	X		
CalibrationFactor									Not required.
CalibrationFactorUnits									Not required.
CalibrationType									Not required.
Coeffa0									Not required.
Coeffa1									Not required.
Coeffa2									Not required.
Coeffa3									Not required.
CoeffOfDetermination									Not required.
CoeffOfDeterminationLimitLow									Not required.
CoeffOfDeterminationLimitType									Not required.
Comment									Not required.
CorrelationCoeff									Not required.
CorrelationCoeffLimitLow									Not required.
CorrelationCoeffLimitType									Not required.
DetectionLimit									Not required.
DetectionLimitType									Not required.
DetectionLimitUnits									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
									Applicability
DifferenceErrorRatio									Not required.
Efficiency									Not required.
Inclusion									Not required.
IntermediateResult	X	X	X	X	X	X	X		If calibrated, report the raw concentration output of the instrument in ug/L for the peak uncorrected for dilution.
IntermediateResultLimitHigh									Not required.
IntermediateResultLimitLow									Not required.
IntermediateResultLimitType									Not required.
IntermediateResultUnits	X	X	X	X	X	X	X		Report "ug/L".
LabQualifiers									Not required.
ManualIntegration									Not required.
Mass	X	X	X	X	X	X	X		For ICP-MS, report the isotope mass.
MassLimitHigh									Not required.
MassLimitLow									Not required.
MassLimitType									Not required.
MassUnits	X	X	X	X	X	X	X		Report "u".
MeanCalibrationFactor									Not required.
MeanCalibrationFactorUnits									Not required.
MeanRetentionTime									Not required.
MeanRetentionTimeLimitHigh									Not required.
MeanRetentionTimeLimitLow									Not required.
MeanRetentionTimeLimitType									Not required.
MeanRetentionTimeLimitUnits									Not required.
MeanRRF									Not required.
MeanRRFLimitLow									Not required.
MeanRRFLimitType									Not required.
PeakID	X	X	X	X	X	X	X		Report a unique identifier. This identifier must be consistent throughout an analytical sequence. For ICP-MS analysis using collision or reaction cell, a "-Gas" suffix must be applied to the PeakID.
PeakRatio									Not required.
PeakRatioLimitHigh									Not required.
PeakRatioLimitLow									Not required.
PeakRatioLimitType									Not required.
PercentDifference									Not required.
PercentDifferenceLimitHigh									Not required.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType									Not required.
PercentRatio	X	X	X	X	X	X	X		For internal standards, report the %RI (Percent Relative Intensity).

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
									Applicability
PercentRatioLimitHigh	X	X	X	X	X	X	X		For internal standards, report the upper limit for the %RI to the nearest whole percent.
PercentRatioLimitLow	X	X	X	X	X	X	X		For internal standards, report the lower limit for the %RI to the nearest whole percent.
PercentRatioLimitType	X	X	X	X	X	X	X		Report "Method".
PercentRecovery									Not required.
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
PercentRSD	X	X	X	X	X	X	X		For ICP, report the %RSD of the replicates to the nearest whole percent.
PercentRSDLimitHigh	X	X	X	X	X	X	X		Report the upper limit for the %RSD to the nearest whole percent.
PercentRSDLimitLow									Not required.
PercentRSDLimitType	X	X	X	X	X	X	X		Report "Method".
QuantitationLimit									Not required.
QuantitationLimitType									Not required.
QuantitationLimitUnits									Not required.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Resolution									Not required.
ResolutionLimitHigh									Not required.
ResolutionLimitLow									Not required.
ResolutionLimitType									Not required.
ResolutionType									Not required.
ResolutionUnits									Not required.
Response	X	X	X	X	X	X	X		Report the mean instrument response output. For internal standards, report the uncorrected intensity.
ResponseLimitHigh									Not required.
ResponseLimitLow									Not required.
ResponseLimitType									Not required.
ResponseType									Not required.
ResponseUnits	X	X	X	X	X	X	X		Report "Peak_Height", "Peak_Area", "Counts", or "Absorbance" as appropriate.
Result									Not required.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType									Not required.

Node and Data Elements	Applicability Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
ResultUncertainty									Not required.
ResultUnits									Not required.
RetentionTime									Not required.
RetentionTimeLimitHigh									Not required.
RetentionTimeLimitLow									Not required.
RetentionTimeLimitType									Not required.
RetentionTimeUnits									Not required.
RRF									Not required.
RRFLimitLow									Not required.
RRFLimitType									Not required.
StandardDeviation									Not required.
StandardDeviationUnits									Not required.
TailingFactor									Not required.
TailingFactorLimitHigh									Not required.
TailingFactorLimitType									Not required.
Wavelength	X	X	X	X	X	X	X		For ICP-AES, Hg, and CN, report the wavelength of the peak in nm.
WavelengthUnits	X	X	X	X	X	X	X		Report "nm".
WeightingFactor									Not required.
PeakComparison	X	X	X	X	X	X	X		
AnalyteName	X	X	X	X	X	X	X		For ICP-MS, report the name of the associated internal standard as it appears in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS number.
ClientAnalyteID	X	X	X	X	X	X	X		Report the CAS number.
ClientAnalyteName									Not required.
Comment									Not required.
LabAnalyteID									Not required.
PeakID	X	X	X	X	X	X	X		Report the unique peak identifier for the associated internal standard.
PeakRatio									Not required.
PeakRatioLimitHigh									Not required.
PeakRatioLimitLow									Not required.
PeakRatioLimitType									Not required.
PercentRatio									Not required.
PercentRatioLimitHigh									Not required.
PercentRatioLimitLow									Not required.
PercentRatioLimitType									Not required.
PeakReplicate	X	X	X	X	X	X	X		
Comment									Not required.
IntermediateResult									Not required.
IntermediateResultLimitHigh									Not required.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
IntermediateResultLimitLow									Not required.
IntermediateResultLimitType									Not required.
IntermediateResultUnits									Not required.
Mass									Not required.
MassLimitHigh									Not required.
MassLimitLow									Not required.
MassLimitType									Not required.
MassUnits									Not required.
PeakReplicateID	X	X	X	X	X	X	X		Report a unique identifier for each replicate.
Resolution									Not required.
ResolutionLimitHigh									Not required.
ResolutionLimitLow									Not required.
ResolutionLimitType									Not required.
ResolutionType									Not required.
ResolutionUnits									Not required.
Response	X	X	X	X	X	X	X		Report the instrument response.
ResponseLimitHigh									Not required.
ResponseLimitLow									Not required.
ResponseLimitType									Not required.
ResponseType									Not required.
ResponseUnits	X	X	X	X	X	X	X		Report "Peak_Height", "Peak_Area", "Counts", or "Absorbance" as appropriate.

| Node and Data Elements | Applicability | | | | | Instructions |
	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_3" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.
SamplePlusMethod						Not required.
InstrumentQC	X	X	X	X	X	
ClientInstrumentQCType						Not required.
ClientMethodCode	X	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID	X	X	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".

	Applicability					
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Comment						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	X	X	Report a unique ID for each QC.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	X	X	Report "RunBatch" for IPC, calibration, ICV, ICB, and ICS. Report "AnalysisBatch" for CCV and CCB.
QCType	X	X	X	X	X	Report "Instrument_Performance_Check_Tune" for Tune; "Initial_Calibration" for calibration; "Initial_Calibration_Verification" for ICV; "Initial_Calibration_Blank" for ICB; "Continuing_Calibration_Verification" for CCV; "Continuing_Calibration_Blank" for CCB; "Interference_Check_Standard_A" for ICSA; or "Interference_Check_Standard_A/B" for ICSAB.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.
LabZipCode	X	X	X	X	X	Report the ZIP or postal code.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
				Applicability		
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisBatch			X	X		Links this analysis to the instrument QC sample that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd			X	X		Links this analysis to the instrument QC sample that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID		X				Links a group of analyses together that are used for the initial calibration. Report the Lab Analysis ID of the standard that starts this calibration sequence.
AnalysisType	X	X	X	X	X	Report "Initial" or "Dilution-01"; then increment as necessary.
Analyst	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount						Not required.
AnalyzedAmountUnits						Not required.
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
BackgroundCorrection	X	X	X	X	X	For ICP-AES and ICP-MS, report "Yes" if background corrections applied; otherwise report "No".
BackgroundRawData	X	X	X	X	X	For ICP-AES and ICP-MS, report "Yes" if background corrections applied before raw data generated. Otherwise report "No".
BackgroundType						Not required.
BottleID						Not required.
ClientAnalysisID						Not required.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Column						Not required.
ColumnInternalDiameter						Not required.
ColumnInternalDiameterUnits						Not required.
ColumnLength						Not required.
ColumnLengthUnits						Not required.
Comment						Not required.
ConfirmationAnalysisID						Not required.
Counts						Not required.
CountsUncertainty						Not required.

	Applicability					
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectorID						Not required.
DetectorType						Not required.
DilutionFactor	X	X	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.
InjectionVolume						Not required.
InjectionVolumeUnits						Not required.
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
InterelementCorrection		X	X	X	X	For ICP-AES and ICP-MS, report "Yes" if interelement corrections were applied; otherwise report "No".
LabAnalysisID	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	Report the lab file ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report month and year the SOW was issued.
OriginalLabAnalysisID						Not required.
PreparationBatch						Not required.
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis						Not required.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
RunBatch	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL (Initial Calibration) sequence.
SampleAmount						Not required.
SampleAmountUnits						Not required.
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.
AnalysisGroup		X				
AnalysisGroupID		X				This links a group of analyses together that are used for the initial calibration. Report the lab analysis ID of the standard that starts this calibration sequence.
AnalysisType		X				Report "Initial_Calibration".
Comment						Not required.
Handling						Not required.
ReportedResult						Not required.
PreparationPlusCleanup		X	X	X		
AliquotAmount		X	X	X		Report the actual amount of standard digested/distilled in mL to at least three significant figures.
AliquotAmountUnits		X	X	X		Report "mL".
Analyst		X	X	X		Report the Analyst's initials.
BottleID						Not required.
CleanedUpDate						Not required.
CleanupBatch						Not required.
CleanupType						Not required.
ClientMethodCode						Not required.
ClientMethodID		X	X	X		Enter the sample preparation ID as described in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource		X	X	X		Report "EPA_CLP".
ClientMethodVersion		X	X	X		Report month and year the SOW was issued.
Comment						Not required.
Efficiency						Not required.
FinalAmount		X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits		X	X	X		Report "mL".

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
InitialAmount						Not required.
InitialAmountUnits						Not required.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
LotNumber						Not required.
MethodCode						Not required.
MethodID		X	X	X		Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource		X	X	X		Report "EPA_CLP".
MethodVersion		X	X	X		Report the month and year the SOW was issued.
PreparationBatch		X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType		X	X	X		Report "Preparation".
PreparationType		X	X	X		Report "Automated" or "Manual".
PreparedDate		X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.
SampleAmount						Not required.
SampleAmountUnits						Not required.
Solvent						Not required.
Characteristic						Not required.
Analyte	X	X	X	X	X	
AmountAdded						Not required.
AmountAddedUnits						Not required.
AmountAddedLocation						Not required.
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	Report "CAS".
AnalyteType	X	X	X	X	X	Report "Target" for all target analytes; "Internal_Standard" for internal standards; "Monitor" for non-target interferences and masses requiring monitoring; or "Instrument_Performance" for tune analytes.
BiasErrorRatio						Not required.
CalibrationBasis		X				Report "Peak" under the AnalysisGroup node.
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.

Node and Data Elements	Applicability					Instructions
	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	
CASRegistryNumber	X	X	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	Report CAS number.
ClientAnalyteName	X	X	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectionLimit		X	X	X	X	Report the current MDL from the default aqueous preparation method or other appropriate method to at least two significant figures.
DetectionLimitType		X	X	X	X	Report "MDL".
DetectionLimitUnits		X	X	X	X	Report "ug/L".
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult		X	X		X	Report the concentration of the standard in ug/L.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits		X	X		X	Report "ug/L".

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
				Applicability		
Inclusion		X				Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult		X	X	X	X	Report the raw concentration output of the instrument unadjusted for sample weight/volume, percent solids, or dilution factor.
IntermediateResultLimitHigh						Not required.
IntermediateResultLimitLow						Not required.
IntermediateResultLimitType						Not required.
IntermediateResultUnits		X	X	X	X	Report "ug/L".
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass						Not required.
MassLimitHigh						Not required.
MassLimitLow						Not required.
MassLimitType						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID		X	X	X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown						Not required.
PercentBreakdownLimitHigh						Not required.
PercentBreakdownLimitType						Not required.
PercentDifference		X				Report the Percent Difference to the nearest whole percent.
PercentDifferenceLimitHigh		X				Report the upper limit for the Percent Difference to the nearest whole percent.
PercentDifferenceLimitLow		X				Report the lower limit for the Percent Difference to the nearest whole percent.
PercentDifferenceLimitType		X				Report "Method".
PercentMatch						Not required.
PercentRecovery			X		X	Report the Percent Recovery to the nearest whole percent. Not required for ICS when true value equals 0.
PercentRecoveryLimitHigh			X		X	Report the upper limit for the Percent Recovery to the nearest whole percent. Not required when ResultLimitHigh applies.
PercentRecoveryLimitLow			X		X	Report the lower limit for the Percent Recovery to the nearest whole percent. Not required when ResultLimitLow applies.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
PercentRecoveryLimitType			X		X	Report "Method".
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationBasis		X				Report "External_Standard" under the AnalysisGroup node.
QuantitationLimit		X	X	X	X	Report the CRQL.
QuantitationLimitType		X	X	X	X	Report "CRQL".
QuantitationLimitUnits		X	X	X	X	Report "ug/L".
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Response						Not required.
ResponseLimitHigh						Not required.
ResponseLimitLow						Not required.
ResponseLimitType						Not required.
ResponseUnits						Not required.
Result		X	X	X	X	For detected target and spike analytes, and for monitored masses, report the final calculated result (in ug/L).
ResultLimitHigh					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.
ResultLimitLow					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.
ResultLimitType					X	Report "Method".
ResultType		X	X	X	X	Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for ICB or CCB results less than the negative MDL (-MDL).
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.
ResultUnits		X	X	X	X	Report "ug/L".
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.
RPDType						Not required.
RRF						Not required.

Applicability

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardConcentration			X	X	X	X	Report the concentration of analyte or internal standard added to the sample in ug/L.
StandardConcentrationUnits			X	X	X	X	Report "ug/L".
StandardDeviation						Not required.
StandardDeviationUnits						Not required.
StandardFinalAmount						Not required.
StandardFinalAmountUnits						Not required.
StandardID	X	X	X	X	X	Report the laboratory-assigned identifier for this standard.
StandardSource	X	X	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
AnalyteComparison		X				
AnalyteName		X				Report the ICP-AES interfering analyte name under the AnalysisGroup node. For ICP-AES target analytes, report as they appear in the SOW.
AnalyteNameContext		X				Report "CAS" under the AnalysisGroup node.
CASRegistryNumber		X				Report the CAS number of the ICP-AES interfering analyte under the AnalysisGroup node. For ICP-AES target analytes, report as they appear in the SOW.
ClientAnalyteID		X				Report the CAS number of the ICP-AES interfering analyte under the AnalysisGroup node.
ClientAnalyteName		X				Report the ICP-AES interfering analyte name under the AnalysisGroup node. For ICP-AES target analytes, report as they appear in the SOW.
Comment						Not required.
CorrectionFactor		X				Enter the ICP-AES interelement correction factor under the AnalysisGroup node to the number of decimal places stored by the instrument and used for correcting the analytical data.
LabAnalyteID						Not required.
AnalyteGroup						Not required.
Peak	X	X	X	X	X	
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType		X				Report "Linear_Regression"; "Linear_Regression_With_Blank_Force"; "Weighted_Linear_Regression"; or "Weighted_Linear_Regression_With_Blank_Force" under the AnalysisGroup node.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
Coeffa0		X				Report the y-intercept of the calibration curve under the AnalysisGroup node.
Coeffa1		X				Report the slope of the calibration curve under the AnalysisGroup node.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff		X				Report the correlation coefficient (r) of the calibration curve to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitLow		X				Report the lower limit for the correlation coefficient to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitType		X				Report "Method" under the AnalysisGroup node.
DetectionLimit						Not required.
DetectionLimitType						Not required.
DetectionLimitUnits						Not required.
DifferenceErrorRatio						Not required.
Efficiency						Not required.
Inclusion		X				Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
IntermediateResult						Not required.
IntermediateResultLimitHigh						Not required.
IntermediateResultLimitLow						Not required.
IntermediateResultLimitType						Not required.
IntermediateResultUnits						Not required.
LabQualifiers						Not required.
ManualIntegration						Not required.
Mass	X	X	X	X	X	For Tune, report the Average Measured Mass. For other ICP-MS analyses, report the isotope mass.
MassLimitHigh	X					For Tune, report the upper limit for the mass.
MassLimitLow	X					For Tune, report the lower limit for the mass.
MassLimitType	X					Report "method".
MassUnits	X	X	X	X	X	Report "u".
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRetentionTime						Not required.
MeanRetentionTimeLimitHigh						Not required.
MeanRetentionTimeLimitLow						Not required.
MeanRetentionTimeLimitType						Not required.
MeanRetentionTimeUnits						Not required.

		Applicability					
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X	X	Report a unique identifier. This identifier must be consistent throughout an analytical sequence. For ICP-MS analysis using collision or reaction cell, a "-Gas" suffix must be applied to the PeakID.
PeakRatio						Not required.
PeakRatioLimitHigh						Not required.
PeakRatioLimitLow						Not required.
PeakRatioLimitType						Not required.
PercentDifference						Not required.
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentRatio		X	X	X	X	For internal standards, report the %RI.
PercentRatioLimitHigh						Not required.
PercentRatioLimitLow						Not required.
PercentRatioLimitType						Not required.
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
PercentRSD	X	X	X	X	X	For ICP, report the %RSD of the replicates to the nearest whole percent.
PercentRSDLimitHigh	X	X	X	X	X	Report the upper limit for the %RSD to the nearest whole percent.
PercentRSDLimitLow						Not required.
PercentRSDLimitType	X	X	X	X	X	Report "Method".
QuantitationLimit						Not required.
QuantitationLimitType						Not required.
QuantitationLimitUnits						Not required.
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Resolution	X					Report the Average Peak Width to at least one decimal place.
ResolutionLimitHigh	X					Report the upper limit from the manufacturer specifications.
ResolutionLimitLow	X					Report the lower limit from the manufacturer specifications.
ResolutionLimitType	X					Report "Laboratory".
ResolutionType						Not required.
ResolutionUnits	X					Report "u".

Applicability

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
Response	X	X	X	X	X	Report the mean instrument response output. For internal standards, report the uncorrected intensity.
ResponseLimitHigh						Not required.
ResponseLimitLow						Not required.
ResponseLimitType						Not required.
ResponseType						Not required.
ResponseUnits	X	X	X	X	X	Report "Peak_Height", "Peak_Area", "Counts", or "Absorbance" as appropriate.
Result						Not required.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType						Not required.
ResultUncertainty						Not required.
ResultUnits						Not required.
RetentionTime						Not required.
RetentionTimeLimitHigh						Not required.
RetentionTimeLimitLow						Not required.
RetentionTimeLimitType						Not required.
RetentionTimeUnits						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardDeviation						Not required.
StandardDeviationUnits						Not required.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength	X	X	X	X	X	For ICP-AES, Hg, and CN, report the wavelength of the peak in nm.
WavelengthUnits	X	X	X	X	X	Report "nm".
WeightingFactor		X				Report "Inverse_Of_Concentration", "Inverse_Square_Of_Concentration", "Variance", "Inverse_Of_Variance", "Standard Deviation", "Inverse_Of_Standard_Deviation", "Inverse_Square_Of_Standard_Deviation", or "None" as applicable under the AnalysisGroup.
PeakComparison		X	X	X	X	
AnalyteName		X	X	X	X	For ICP-MS, report the name of the associated internal standard as it appears in the SOW.
AnalyteNameContext		X	X	X	X	Report "CAS".
CASRegistryNumber		X	X	X	X	Report the CAS number.
ClientAnalyteID		X	X	X	X	Report the CAS number.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
ClientAnalyteName		X	X	X	X	Report the analytes as they appear in the SOW.
Comment						Not required.
PeakID		X	X	X	X	Report the unique peak identifier of the associated internal standard.
PeakRatio						Not required.
PeakRatioLimitHigh						Not required.
PeakRatioLimitLow						Not required.
PeakRatioLimitType						Not required.
PercentRatio						Not required.
PercentRatioLimitHigh						Not required.
PercentRatioLimitLow						Not required.
PercentRatioLimitType						Not required.
PeakReplicate	X	X	X	X	X	
Comment						Not required.
IntermediateResult						Not required.
IntermediateResultLimitHigh						Not required.
IntermediateResultLimitLow						Not required.
IntermediateResultUnits						Not required.
Mass						Not required.
MassLimitHigh						Not required.
MassLimitLow						Not required.
MassLimitType						Not required.
MassUnits						Not required.
PeakReplicateID	X	X	X	X	X	Report a unique identifier for each replicate.
Resolution						Not required.
ResolutionLimitHigh						Not required.
ResolutionLimitLow						Not required.
ResolutionLimitType						Not required.
ResolutionType						Not required.
ResolutionUnits						Not required.
Response	X	X	X	X	X	Report the instrument response.
ResponseLimitHigh						Not required.
ResponseLimitLow						Not required.
ResponseLimitType						Not required.
ResponseType						Not required.
ResponseUnits	X	X	X	X	X	Report "Peak_Height", "Peak_Area", "Counts", or "Absorbance" as applicable.

7.2   Stage 2b

TABLE 2. INORGANICS DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
Header	X	X	X	X	X	X	X	X	
ClientID	X	X	X	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName									Not required.
Comment									Not required.
DateFormat	X	X	X	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabDataPackageID	X	X	X	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabNarrative									Not required.
LabQualifiersDefinition	X	X	X	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	X	X	X	Report the Case Number.
ProjectName									Not required.
SiteID									Not required.
SiteName									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
SamplePlusMethod	X	X	X	X	X	X	X	X	
ClientID	X	X	X						Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory									Not required.
ClientMethodCode	X	X	X	X	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID	X	X	X	X	X	X	X		Report the Modified Analysis Number, if applicable
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
ClientMethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName									Not required.
ClientSampleID	X	X	X	X	X	X	X	X	Report the EPA Sample Number.
CollectedDate	X	X	X						Report the date and time the sample was collected.
CollectedEndDate									Not required.
Comment									Not required.
Composite									Not required.
CoolerID									Not required.
CustodyID	X								Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch									Not required.
Filtered	X								Report "Yes" for dissolved metals, or "No" for total metals.
LabContract	X	X	X	X	X	X	X		Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID									Not required.
LabMethodName									Not required.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X	X						Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID									Not required.
LocationName									Not required.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
MatrixID	X	X	X	X	X	X	X	X	Report "Water", "Soil", or "Wipe" as applicable.
MatrixMedium	X	X	X	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable. Use "Solid" for wipes.
MethodBatch									Not required.
MethodCategory									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
MethodLevel									Not required.
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X	X			X	X		Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID									Not required.
PhaseAnalyzed									Not required.
Preservative	X	X	X						Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X	X	X	X		Report the Case Number.
ProjectName									Not required.
QCCategory		X	X	X	X	X	X		Report "Blank" for PB and LEB; "Spike" for MS and post-digestion spike; "Blank_Spike" for LCS; "Duplicate" for duplicate; or "Serial_Dilution" for SD.
QCLinkage		X	X	X	X	X	X		Report "LabReportingBatch" for MS, post-digestion spike, Dup, and SD; or "PreparationBatch" for PB and LCS.
QCType	X	X	X	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "PT_Sample" for Performance Evaluation samples or Proficiency Testing audit samples; "Method_Blank" for PB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Duplicate" for Dup; "Laboratory_Control_Sample" for LCS; "Post_Digestion_Spike" for post-digestion spikes; "Serial_Dilution" for SD; or "Non_Client_Sample" for NCS.
Quarantine	X								Report "Yes" or "No" based on sampling information.
SamplingBatch									Not required.
ShippingBatch									Not required.
SiteID									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
SiteName									Not required.
StorageBatch									Not required.
InstrumentQC									Not required.
Characteristic	X	X	X	X	X	X	X		
CharacteristicType	X	X	X	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH". Wipe samples do not require "Percent_Solids", "pH", or "Temperature".
CharacteristicValue	X	X	X	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X	X	X	X		Report "C" for "Temperature".
Comment									Not required.
ContactInformation	X	X	X	X	X	X	X	X	
LabAddress1	X	X	X	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType									Not required.
LabState	X	X	X	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	X	X	X	Report the 10-digit phone number for the laboratory.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
LabType									Not required.
LabZipCode	X	X	X	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	X	X	X	
AliquotAmount									Not required.
AliquotAmountUnits									Not required.
AnalysisBatch	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd	X	X	X	X	X	X	X	X	Links this analysis to the instrument QC sample(s) that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration									Not required.
AnalysisDurationUnits									Not required.
AnalysisGroupID									Not required.
AnalysisType	X	X	X	X	X	X	X		Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.
Analyst	X	X	X	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount									Not required.
AnalyzedAmountUnits									Not required.
AnalyzedDate	X	X	X	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
Column									Not required.
ColumnInternalDiameter									Not required.
ColumnInternalDiameterUnits									Not required.
ColumnLength									Not required.
ColumnLengthUnits									Not required.
Comment									Not required.
ConfirmationAnalysisID									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
				Applicability					
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectorID									Not required.
DetectorType									Not required.
DilutionFactor	X	X	X	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency									Not required.
HeatedPurge									Not required.
Inclusion									Not required.
InjectionVolume									Not required.
InjectionVolumeUnits									Not required.
InstrumentID	X	X	X	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	X	X	X	Report the lab file ID.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch									Not required.
ProcedureID									Not required.
ProcedureName									Not required.
ReferenceDate									Not required.
ResultBasis	X	X	X		X				Report "Dry" for soil/sediment samples. For aqueous/water samples, report "Dissolved" if sample field-filtered; otherwise report "Total". Report "Wet" for tissue samples or for any other matrices (not aqueous/water) for which the results are not corrected for percent solids.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
RunBatch	X	X	X	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL (Initial Calibration) sequence.
Temperature									Not required.
TemperatureUnits									Not required.
Wavelength									Not required.
WavelengthUnits									Not required.
Yield									Not required.
AnalysisGroup									Not required.
Handling									Not required.
ReportedResult	X	X	X	X	X	X	X		
AnalysisGroupID									Not required.
AnalyteGroupID	X	X	X	X	X	X	X		Report the unique identifier from the AnalyteGroup the Hardness result is derived from.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness or "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS analyses. Report "Derived" for Hardness.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Numbers as they appear in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit									Not required.
ClientDetectionLimitUnits									Not required.
ClientQuantitationLimit	X	X	X	X	X	X	X		Report the unadjusted CRQL.
ClientQuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
Comment									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures. Not required for Hardness.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
ExpectedResult		X		X		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes or the true value for LCS.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits		X		X		X			Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water (or "mg/L" for TCLP), or "ug" for wipe samples.
LabAnalysisID	X	X	X	X	X	X	X		Report the unique identifier from the analysis this reported result was derived from. Not required for Hardness.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-IN.
LabResultStatus	X	X	X						Report "Preliminary" or "Final" as applicable.
PeakID									Not required.
PercentDifference							X		Report the Percent Difference to the nearest whole percent.
PercentDifferenceLimitHigh							X		Report the upper limit for the Percent Difference to the nearest whole percent.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType							X		Report "Method".
PercentRecovery		X		X		X			Report the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitHigh		X		X					Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow		X		X					Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X		X					Report "Method".
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
Result	X	X	X	X	X	X	X		Report the final calculated result for detects per the SOW.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
RetentionTime									Not required.
RetentionTimeUnits									Not required.
RPD			X						Report the RPD to the nearest whole percent.
RPDLimitHigh			X						Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType			X						Report "Method".
RPDType									Not required.
PreparationPlusCleanup	X	X	X	X	X	X	X		
AliquotAmount	X	X	X	X	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water to at least three significant figures. Not required for wipes.
AliquotAmountUnits	X	X	X	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water. Not required for wipes.
Analyst	X	X	X	X	X	X	X		Report the Analyst's initials.
CleanedUpDate									Not required.
CleanUpBatch									Not required.
CleanUpType									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X		Report the sample preparation ID as given in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.

Node and Data Elements	Applicability Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
ClientMethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X		Report the month and year the SOW was issued.
Comment									Not required.
FinalAmount	X	X	X	X	X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits	X	X	X	X	X	X	X		Report "mL".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
LotNumber									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X		Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X	X	X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType	X	X	X	X	X	X	X		Report "Preparation".
PreparationType	X	X	X	X	X	X	X		Report "Automated" or "Manual".
PreparedDate	X	X	X	X	X	X	X		Report the date and time the sample was prepared.
ProcedureID									Not required.
ProcedureName									Not required.
Solvent									Not required.
Analyte	X	X	X	X	X	X	X		
AnalyteGroupID	X	X	X	X	X	X	X		Report the identifier that links the Ca or Mg result to the AnalyteGroup Hardness result.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness; "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS; "Internal_Standard" for internal standards; or "Monitor" for non-target interferences and masses requiring monitoring.

Node and Data Elements	Applicability								Instructions
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
BiasErrorRatio									Not required.
CalibrationBasis									Not required.
CalibrationFactor									Not required.
CalibrationFactorUnits									Not required.
CalibrationType									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
Coeffa0									Not required.
Coeffa1									Not required.
Coeffa2									Not required.
Coeffa3									Not required.
CoeffOfDetermination									Not required.
CoeffOfDeterminationLimitLow									Not required.
CoeffOfDeterminationLimitType									Not required.
Comment									Not required.
CorrelationCoeff									Not required.
CorrelationCoeffLimitLow									Not required.
CorrelationCoeffLimitType									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.
Efficiency									Not required.
ExpectedResult									Not required.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits									Not required.
Inclusion									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report the qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard (Internal Standards and spiking analytes only).
Mass									Not required.
MassUnits									Not required.
MeanCalibrationFactor									Not required.
MeanCalibrationFactorUnits									Not required.
MeanRRF									Not required.
MeanRRFLimitLow									Not required.
MeanRRFLimitType									Not required.
PeakID	X	X	X	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown									Not required.
PercentBreakdownLimitHigh									Not required.
PercentBreakdownLimitType									Not required.
PercentDifference									Not required.
PercentDifferenceLimitHigh									Not required.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType									Not required.
PercentRecovery									Not required.
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
PercentRSD									Not required.
PercentRSDLimitHigh									Not required.
PercentRSDLimitLow									Not required.
PercentRSDLimitType									Not required.
QuantitationBasis									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.

| Node and Data Elements | Applicability | | | | | | | Instructions |
	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		For target or spike, analyte detects, and for monitored masses, report the final calculated result.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, and "ug" for wipe samples.
RPD									Not required.
RPDLimitHigh									Not required.
RPDLimitType									Not required.
RPDType									Not required.
RRF									Not required.
RRFLimitLow									Not required.
RRFLimitType									Not required.
StandardSource	X	X	X	X	X	X	X		Report the vendor/manufacturer for this standard.
TailingFactor									Not required.
TailingFactorLimitHigh									Not required.
TailingFactorLimitType									Not required.
Wavelength									Not required.
WavelengthUnits									Not required.
WeightingFactor									Not required.
AnalyteGroup	X	X	X	X	X	X	X		
AnalyteGroupID	X	X	X	X	X	X	X		Report a unique identifier.
AnalyteName	X	X	X	X	X	X	X		Report "Hardness".

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Derived".
CASRegistryNumber	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteID	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteName	X	X	X	X	X	X	X		Report "Hardness".
Comment									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW. Include the Q qualifiers from Form 1-IN.
Result	X	X	X	X	X	X	X		Report the final calculated for detects per the SOW.
ResultType	X	X	X	X	X	X	X		Report "=" for detects. Report "Not_Detected" for non-detects (where both Ca and Mg are not detected).
ResultUncertainty									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/L".
Peak									Not required.
PeakComparison									Not required.

	Applicability					
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
Header	X	X	X	X	X	
ClientID	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName						Not required.
Comment						Not required.
DateFormat	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2b" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	Report "3" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	Report the software version number.
LabContract	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription						Not required.
LabContractModificationID						Not required.
LabDataPackageID	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabNarrative						Not required.
LabQualifiersDefinition	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	Report the Case Number.
ProjectName						Not required.
SiteID						Not required.
SiteName						Not required.
SamplePlusMethod						Not required.
InstrumentQC	X	X	X	X	X	
ClientInstrumentQCType						Not required.
ClientMethodCode	X	X	X	X	X	Report "TCLP" or "SPLP" when applicable.
ClientMethodID	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID	X	X	X	X	X	Report the Modified Analysis Number, if applicable.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Comment						Not required.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabInstrumentQCID	X	X	X	X	X	Report a unique ID for each QC.
LabMethodID						Not required.
LabMethodName						Not required.
LabName	X	X	X	X	X	Report the Lab Name.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
QCLinkage	X	X	X	X	X	Report "RunBatch" for IPC, calibration, ICV, ICB, and ICS. Report "AnalysisBatch" for CCV and CCB.
QCType	X	X	X	X	X	Report "Instrument_Performance_Check_Tune" for Tune; "Initial_Calibration" for calibration; "Initial_Calibration_Verification" for ICV; "Initial_Calibration_Blank" for ICB; "Continuing_Calibration_Verification" for CCV; "Continuing_Calibration_Blank" for CCB; "Interference_Check_Standard_A" for ICSA; or "Interference_Check_Standard_A/B" for ICSAB.
ContactInformation	X	X	X	X	X	
LabAddress1	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	Report the name of person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType						Not required.
LabState	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType						Not required.
LabZipCode	X	X	X	X	X	Report the ZIP or postal code.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
Analysis	X	X	X	X	X	
AliquotAmount						Not required.
AliquotAmountUnits						Not required.
AnalysisBatch			X	X		Links this analysis to the instrument QC sample that begins this sequence. Report the Lab Analysis ID of the CCV that starts the sequence.
AnalysisBatchEnd			X	X		Links this analysis to the instrument QC sample that ends this sequence. Report the Lab Analysis ID of the CCV that ends this sequence.
AnalysisDuration						Not required.
AnalysisDurationUnits						Not required.
AnalysisGroupID		X				Links a group of analyses together that are used for the initial calibration. Report the Lab Analysis ID of the standard that starts this calibration sequence.
AnalysisType	X	X	X	X	X	Report "Initial" or "Dilution-01"; then increment as necessary.
Analyst	X	X	X	X	X	Report the Analyst's initials.
AnalyzedAmount						Not required.
AnalyzedAmountUnits						Not required.
AnalyzedDate	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID						Not required.
ClientMethodCode						Not required.
ClientMethodID	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
Column						Not required.
ColumnInternalDiameter						Not required.
ColumnInternalDiameterUnits						Not required.
ColumnLength						Not required.
ColumnLengthUnits						Not required.
Comment						Not required.
ConfirmationAnalysisID						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
CountsUnits						Not required.
DetectorID						Not required.
DetectorType						Not required.
DilutionFactor	X	X	X	X	X	Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency						Not required.
HeatedPurge						Not required.
Inclusion						Not required.
InjectionVolume						Not required.
InjectionVolumeUnits						Not required.
InstrumentID	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	Report the lab file ID.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
MethodCode						Not required.
MethodID	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.
MethodSource	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch						Not required.
ProcedureID						Not required.
ProcedureName						Not required.
ReferenceDate						Not required.
ResultBasis						Not required.
RunBatch	X	X	X	X	X	Links this analysis to an initial calibration. Report the Lab Analysis ID of the standard (Tune or calibration standard) that started the ICAL (Initial Calibration) sequence.
Temperature						Not required.
TemperatureUnits						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
Yield						Not required.

		Applicability				

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
AnalysisGroup		X				
AnalysisGroupID		X				This links a group of analyses together that are used for the initial calibration. Report the lab analysis ID of the standard that starts this calibration sequence.
AnalysisType		X				Report "Initial_Calibration".
Comment						Not required.
Handling						Not required.
ReportedResult						Not required.
PreparationPlusCleanup		X	X	X		
AliquotAmount		X	X	X		Report the actual amount of standard digested/distilled in mL to at least three significant figures.
AliquotAmountUnits		X	X	X		Report "mL".
Analyst		X	X	X		Report the Analyst's initials.
CleanedUpDate						Not required.
CleanupBatch						Not required.
CleanupType						Not required.
ClientMethodCode						Not required.
ClientMethodID		X	X	X		Enter the sample preparation ID as described in Exhibit B – Reporting and Deliverables Requirements.
ClientMethodModificationDescription						Not required.
ClientMethodModificationID						Not required.
ClientMethodName						Not required.
ClientMethodSource		X	X	X		Report "EPA_CLP".
ClientMethodVersion		X	X	X		Report the month and year the SOW was issued.
Comment						Not required.
FinalAmount		X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits		X	X	X		Report "mL".
InitialAmount						Not required.
InitialAmountUnits						Not required.
LabID						Not required.
LabMethodID						Not required.
LabMethodName						Not required.
LabName						Not required.
LotNumber						Not required.
MethodCode						Not required.
MethodID		X	X	X		Report "ISM02.4".
MethodModificationDescription						Not required.
MethodModificationID						Not required.
MethodName						Not required.

	Applicability					Instructions
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	
MethodSource		X	X	X		Report "EPA_CLP".
MethodVersion		X	X	X		Report the month and year the SOW was issued.
PreparationBatch		X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType		X	X	X		Report "Preparation".
PreparationType		X	X	X		Report "Automated" or "Manual".
PreparedDate		X	X	X		Report the date and time the sample was prepared.
ProcedureID						Not required.
ProcedureName						Not required.
Solvent						Not required.
Characteristic						Not required.
Analyte	X	X	X	X	X	
AnalyteGroupID						Not required.
AnalyteName	X	X	X	X	X	Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	Report "CAS".
AnalyteType	X	X	X	X	X	Report "Target" for all target analytes; "Internal_Standard" for internal standards; "Monitor" for non-target interferences and masses requiring monitoring; or "Instrument_Performance" for tune analytes.
BiasErrorRatio						Not required.
CalibrationBasis		X				Report "Peak" under the AnalysisGroup node.
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType						Not required.
CASRegistryNumber	X	X	X	X	X	Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	Report CAS number.
ClientAnalyteName	X	X	X	X	X	Report the analytes as they appear in the SOW.
Coeffa0						Not required.
Coeffa1						Not required.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff						Not required.
CorrelationCoeffLimitLow						Not required.
CorrelationCoeffLimitType						Not required.
Counts						Not required.
CountsUncertainty						Not required.
CountsUncertaintyConfidenceLevel						Not required.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
			Applicability			
CountsUncertaintyDetermination						Not required.
CountsUncertaintyIntervalType						Not required.
CountsUncertaintyLimitHigh						Not required.
CountsUncertaintyLimitLow						Not required.
CountsUncertaintyType						Not required.
CountsUnits						Not required.
DetectionLimit		X	X	X	X	Report the current MDL from the default aqueous preparation method or other appropriate method to at least two significant figures.
DetectionLimitType		X	X	X	X	Report "MDL".
DetectionLimitUnits		X	X	X	X	Report "ug/L".
DifferenceErrorRatio						Not required.
Efficiency						Not required.
ExpectedResult		X	X		X	Report the concentration of the standard in ug/L.
ExpectedResultUncertainty						Not required.
ExpectedResultUncertaintyConfidenceLevel						Not required.
ExpectedResultUncertaintyDetermination						Not required.
ExpectedResultUncertaintyIntervalType						Not required.
ExpectedResultUncertaintyLimitHigh						Not required.
ExpectedResultUncertaintyLimitLow						Not required.
ExpectedResultUncertaintyType						Not required.
ExpectedResultUncertaintyUnits						Not required.
ExpectedResultUnits		X	X		X	Report "ug/L".
Inclusion		X				Report "No" if an analyte in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabAnalyteID						Not required.
LabQualifiers	X	X	X	X	X	Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	Report the vendor/manufacturer-assigned lot number for this standard.
Mass						Not required.
MassUnits						Not required.
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID		X	X	X	X	If response from a single peak is used for quantitation, report the ID of that peak.
PercentBreakdown						Not required.
PercentBreakdownLimitHigh						Not required.
PercentBreakdownLimitType						Not required.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
PercentDifference		X				Report the Percent Difference to the nearest whole percent.
PercentDifferenceLimitHigh		X				Report the upper limit for the Percent Difference.
PercentDifferenceLimitLow		X				Report the lower limit for the Percent Difference.
PercentDifferenceLimitType		X				Report "Method".
PercentRecovery			X		X	Report the Percent Recovery to the nearest whole percent. Not required for ICS when true value equals 0.
PercentRecoveryLimitHigh			X		X	Report the upper limit for the Percent Recovery to the nearest whole percent. Not required when ResultLimitHigh applies.
PercentRecoveryLimitLow			X		X	Report the lower limit for the Percent Recovery to the nearest whole percent. Not required when ResultLimitLow applies.
PercentRecoveryLimitType			X		X	Report "Method".
PercentRecoveryType						Not required.
PercentRSD						Not required.
PercentRSDLimitHigh						Not required.
PercentRSDLimitLow						Not required.
PercentRSDLimitType						Not required.
QuantitationBasis		X				Report "External_Standard" under the AnalysisGroup node.
QuantitationLimit		X	X	X	X	Report the CRQL.
QuantitationLimitType		X	X	X	X	Report "CRQL".
QuantitationLimitUnits		X	X	X	X	Report "ug/L".
ReportingLimit						Not required.
ReportingLimitType						Not required.
ReportingLimitUnits						Not required.
Result		X	X	X	X	For detected target and spike analytes, and for monitored masses, report the final calculated result (in ug/L).
ResultLimitHigh					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.
ResultLimitLow					X	For analytes and interferents with true values less than 5x (10x for ICP-MS) CRQL.
ResultLimitType					X	Report "Method".
ResultType		X	X	X	X	Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for ICB or CCB results less than the negative MDL (-MDL).
ResultUncertainty						Not required.
ResultUncertaintyConfidenceLevel						Not required.
ResultUncertaintyDetermination						Not required.
ResultUncertaintyIntervalType						Not required.
ResultUncertaintyLimitHigh						Not required.
ResultUncertaintyLimitLow						Not required.
ResultUncertaintyType						Not required.
ResultUncertaintyUnits						Not required.

		Applicability				
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
ResultUnits		X	X	X	X	Report "ug/L".
RPD						Not required.
RPDLimitHigh						Not required.
RPDLimitType						Not required.
RPDType						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
StandardSource	X	X	X	X	X	Report the vendor/manufacturer for this standard.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength						Not required.
WavelengthUnits						Not required.
WeightingFactor						Not required.
AnalyteGroup						Not required.
Peak	X	X	X	X	X	
CalibrationFactor						Not required.
CalibrationFactorUnits						Not required.
CalibrationType		X				Report "Linear_Regression"; "Linear_Regression_With_Blank_Force"; "Weighted_Linear_Regression"; or "Weighted_Linear_Regression_With_Blank_Force" under the AnalysisGroup node.
Coeffa0		X				Report the y-intercept of the calibration curve under the AnalysisGroup node.
Coeffa1		X				Report the slope of the calibration curve under the AnalysisGroup node.
Coeffa2						Not required.
Coeffa3						Not required.
CoeffOfDetermination						Not required.
CoeffOfDeterminationLimitLow						Not required.
CoeffOfDeterminationLimitType						Not required.
Comment						Not required.
CorrelationCoeff		X				Report the correlation coefficient (r) of the calibration curve to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitLow		X				Report the lower limit for the correlation coefficient to at least 4 significant figures under the AnalysisGroup node.
CorrelationCoeffLimitType		X				Report "Method" under the AnalysisGroup node.
DifferenceErrorRatio						Not required.
Efficiency						Not required.

	Applicability					
Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
Inclusion		X				Report "No" if a peak in a standard is not to be included in the calibration curve; otherwise report "Yes".
LabQualifiers						Not required.
Mass	X					Report the Average Measured Mass.
MassLimitHigh	X					Report the upper limit for the mass.
MassLimitLow	X					Report the lower limit for the mass.
MassLimitType	X					Report "method".
MassUnits	X					Report "u".
MeanCalibrationFactor						Not required.
MeanCalibrationFactorUnits						Not required.
MeanRetentionTime						Not required.
MeanRetentionTimeLimitHigh						Not required.
MeanRetentionTimeLimitLow						Not required.
MeanRetentionTimeLimitType						Not required.
MeanRetentionTimeUnits						Not required.
MeanRRF						Not required.
MeanRRFLimitLow						Not required.
MeanRRFLimitType						Not required.
PeakID	X	X	X	X	X	Report a unique identifier. This identifier must be consistent throughout an analytical sequence. For ICP-MS analysis using collision or reaction cell, a "-Gas" suffix must be applied to the PeakID.
PercentDifference						Not required.
PercentDifferenceLimitHigh						Not required.
PercentDifferenceLimitLow						Not required.
PercentDifferenceLimitType						Not required.
PercentRecovery						Not required.
PercentRecoveryLimitHigh						Not required.
PercentRecoveryLimitLow						Not required.
PercentRecoveryLimitType						Not required.
PercentRecoveryType						Not required.
PercentRSD	X	X	X	X	X	For ICP, report the %RSD of the replicates to the nearest whole percent.
PercentRSDLimitHigh	X	X	X	X	X	Report the upper limit for the %RSD to the nearest whole percent.
PercentRSDLimitLow						Not required.
PercentRSDLimitType	X	X	X	X	X	Report "Method".
Resolution	X					Report the Average Peak Width to at least one decimal place.
ResolutionLimitHigh	X					Report the upper limit from the manufacturer specifications.
ResolutionLimitLow	X					Report the lower limit from the manufacturer specifications.
ResolutionLimitType	X					Report "Laboratory".
ResolutionType						Not required.

Node and Data Elements	IPC	ICAL	ICV/CCV	ICB/CCB	ICS	Instructions
						Applicability
ResolutionUnits	X					Report "u".
Result						Not required.
ResultLimitHigh						Not required.
ResultLimitLow						Not required.
ResultLimitType						Not required.
ResultType						Not required.
ResultUncertainty						Not required.
ResultUnits						Not required.
RRF						Not required.
RRFLimitLow						Not required.
RRFLimitType						Not required.
TailingFactor						Not required.
TailingFactorLimitHigh						Not required.
TailingFactorLimitType						Not required.
Wavelength	X	X	X	X	X	For ICP-AES, Hg, and CN, report the wavelength of the peak in nm.
WavelengthUnits	X	X	X	X	X	Report "nm".
WeightingFactor		X				Report "Inverse_Of_Concentration", "Inverse_Square_Of_Concentration", "Variance", "Inverse_Of_Variance", "Standard_Deviation", "Inverse_Of_Standard_Deviation", "Inverse_Square_Of_Standard_Deviation", or "None" as applicable under the AnalysisGroup node.
PeakComparison		X	X	X	X	
Comment						Not required.
PeakID		X	X	X	X	Report the unique peak identifier of the associated internal standard.
PercentRatio						Not required.
PercentRatioLimitHigh						Not required.
PercentRatioLimitLow						Not required.
PercentRatioLimitType						Not required.

7.3   Stage 2a

TABLE 3. INORGANICS DATA ELEMENT INSTRUCTIONS

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
Header	X	X	X	X	X	X	X	X	
ClientID	X	X	X	X	X	X	X	X	Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientName									Not required.
Comment									Not required.
DateFormat	X	X	X	X	X	X	X	X	Report MMDDYYYYThh:mm:ss. All dates and times reported in the EDD must follow this format. If any part of the time is unknown, report "00" for the unknown hours, minutes, and seconds.
EDDID	X	X	X	X	X	X	X	X	Report "SEDD".
EDDImplementationID	X	X	X	X	X	X	X	X	Report "SEDD_5-2_GENERAL_2a" (This is the DTD used).
EDDImplementationVersion	X	X	X	X	X	X	X	X	Report "2" (This is the version of the DTD used).
EDDVersion	X	X	X	X	X	X	X	X	Report "5.2".
GeneratingSystemID	X	X	X	X	X	X	X	X	Report the name of generating software or vendor.
GeneratingSystemVersion	X	X	X	X	X	X	X	X	Report the software version number.
Lab Contract	X	X	X	X	X	X	X	X	Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabDataPackageID	X	X	X	X	X	X	X	X	Report the SDG.
LabDataPackageName	X	X	X	X	X	X	X	X	Report "ICP_AES", "ICP_MS", "Hg", or "CN" as applicable.
LabDataPackageVersion	X	X	X	X	X	X	X	X	Report "1", then increment with each resubmission.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
Lab Name	X	X	X	X	X	X	X	X	Report the Lab Name.
LabNarrative									Not required.
LabQualifiersDefinition	X	X	X	X	X	X	X	X	Use the format 'Qualifier:Definition' to report each qualifier used. Use a ';' to separate the definitions of multiple qualifiers.
LabReportedDate	X	X	X	X	X	X	X	X	Report the date this data was reported to the client.
ProjectID	X	X	X	X	X	X	X	X	Report the Case Number.
ProjectName									Not required.
SiteID									Not required.
SiteName									Not required.
SamplePlusMethod	X	X	X	X	X	X	X	X	
ClientID	X	X	X						Report "1" for Region 1, "2" for Region 2, etc. For samples received from QATS, report "91".
ClientMethodCategory									Not required.
ClientMethodCode	X	X	X	X	X	X	X	X	Report "TCLP" or "SPLP" when applicable.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID	X	X	X	X	X	X	X		Report the Modified Analysis Number, if applicable.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
ClientMethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
ClientName									Not required.
ClientSampleID	X	X	X	X	X	X	X	X	Report the EPA Sample Number.
CollectedDate	X	X	X						Report the date and time the sample was collected.
CollectedEndDate									Not required.
Comment									Not required.
Composite									Not required.
CoolerID									Not required.
CustodyID	X								Report the Traffic Report/Chain of Custody Record Form number.
EquipmentBatch									Not required.
Filtered	X								Report "Yes" for dissolved metals, or "No" for total metals.
LabContract	X	X	X	X	X	X	X		Report the Contract Number.
LabContractModificationDescription									Not required.
LabContractModificationID									Not required.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabMethodID									Not required.
LabMethodName									Not required.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabReceiptDate	X	X	X						Report the date and time the sample was received.
LabReportingBatch	X	X	X	X	X	X	X	X	Links all samples analyzed to this deliverable. Report the SDG Number.
LabSampleID	X	X	X	X	X	X	X	X	Report the Lab Sample ID as assigned by the laboratory.
LocationID									Not required.
LocationName									Not required.
MatrixID	X	X	X	X	X	X	X	X	Report "Water", "Soil", or "Wipe" as applicable.
MatrixMedium	X	X	X	X	X	X	X	X	Report "Aqueous" or "Solid" as applicable. Use "Solid" for wipes.
MethodBatch									Not required.
MethodCategory									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
MethodLevel									Not required.
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodType	X	X	X	X	X	X	X	X	Report "ICP/AES", "ICP/MS", "CVAA", or "Spectrophotometry" as applicable.
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
OriginalClientSampleID		X	X			X	X		Report the EPA Sample Number of the original sample this sample was derived from.
OriginalLabSampleID									Not required.
PhaseAnalyzed									Not required.
Preservative	X	X	X						Report any chemical or physical preservative used. Report "None" if sample was not preserved.
ProjectID	X	X	X	X	X	X	X		Report the Case Number.
ProjectName									Not required.
QCCategory		X	X	X	X	X	X		Report "Blank" for PB and LEB; "Spike" for MS and post-digestion spike; "Blank_Spike" for LCS; "Duplicate" for duplicate; or "Serial_Dilution" for SD.
QCLinkage		X	X	X	X	X	X		Report "LabReportingBatch" for MS, post-digestion spike, Dup, and SD; or "PreparationBatch" for PB and LCS.
QCType	X	X	X	X	X	X	X	X	Report "Field_Sample" for field samples; "Field_Blank" for field, equipment, rinse, or trip blanks; "PT_Sample" for Performance Evaluation samples or Proficiency Testing audit samples; "Method_Blank" for PB; "Leachate_Extraction_Blank" for LEB; "Matrix_Spike" for MS; "Duplicate" for Dup; "Laboratory_Control_Sample" for LCS; "Post_Digestion_Spike" for post-digestion spikes; "Serial_Dilution" for SD; or "Non_Client_Sample" for NCS.
Quarantine	X								Report "Yes" or "No" based on sampling information.
SamplingBatch									Not required.
ShippingBatch									Not required.
SiteID									Not required.
SiteName									Not required.
StorageBatch									Not required.
Characteristic	X	X	X	X	X	X	X		
CharacteristicType	X	X	X	X	X	X	X		Report "Percent_Solids" for each SamplePlusMethod. Report "pH" and "Temperature" for samples, received at the laboratory, under each SamplePlusMethod node. Tissue samples do not require "Percent_Solids" or "pH". Wipe samples do not require "Percent_Solids", "pH", or "Temperature".

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
CharacteristicValue	X	X	X	X	X	X	X		For "Percent_Solids", report "0.0" for aqueous/water samples including QC samples; report the percent solids to two significant figures if less than 10 and three significant figures if greater than or equal to 10 for soil/sediment samples including QC samples. For "pH", report the pH to the nearest tenth for aqueous/water samples (and soil/sediment samples as requested). For "Temperature", report the temperature at receipt to the nearest degree for aqueous/water and soil/sediment samples received at the laboratory.
CharacteristicUnits	X	X	X	X	X	X	X		Report "C" for "Temperature".
Comment									Not required.
ContactInformation	X	X	X	X	X	X	X	X	
LabAddress1	X	X	X	X	X	X	X	X	Report the street address of the laboratory.
LabAddress2	X	X	X	X	X	X	X	X	If applicable, report any additional address information (e.g., suite, maildrop). Otherwise leave blank.
LabCity	X	X	X	X	X	X	X	X	Report the city in which the laboratory is located.
LabCountry	X	X	X	X	X	X	X	X	Report the country in which the laboratory is located.
LabID	X	X	X	X	X	X	X	X	Report the Agency-assigned Lab Code.
LabName	X	X	X	X	X	X	X	X	Report the Lab Name.
LabPointOfContact	X	X	X	X	X	X	X	X	Report the name of the person at the laboratory serving as the point of contact.
LabPointOfContactElectronicAddress	X	X	X	X	X	X	X	X	Report the Email address of the point of contact.
LabPointOfContactTitle	X	X	X	X	X	X	X	X	Report the title of the point of contact.
LabPointOfContactType									Not required.
LabState	X	X	X	X	X	X	X	X	Report the state or province in which the laboratory is located.
LabTelephoneNumber	X	X	X	X	X	X	X	X	Report the 10-digit phone number for the laboratory.
LabType									Not required.
LabZipCode	X	X	X	X	X	X	X	X	Report the ZIP or postal code.
Analysis	X	X	X	X	X	X	X	X	
AliquotAmount									Not required.
AliquotAmountUnits									Not required.
AnalysisDuration									Not required.
AnalysisDurationUnits									Not required.
AnalysisGroupID									Not required.
AnalysisType	X	X	X	X	X	X	X		Report "Initial", "Dilution-01", or "Reanalysis-01"; then increment as necessary.
Analyst	X	X	X	X	X	X	X	X	Report the Analyst's initials.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
AnalyzedAmount									Not required.
AnalyzedAmountUnits									Not required.
AnalyzedDate	X	X	X	X	X	X	X	X	Report the date and time the sample was analyzed.
ClientAnalysisID									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X	X	Report month and year the SOW was issued.
Column									Not required.
ColumnInternalDiameter									Not required.
ColumnInternalDiameterUnits									Not required.
ColumnLength									Not required.
ColumnLengthUnits									Not required.
Comment									Not required.
ConfirmationAnalysisID									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectorID									Not required.
DetectorType									Not required.
DilutionFactor	X	X	X	X	X	X	X		Report the Dilution Factor used to the nearest tenth. Report "1.0" when no dilutions are used.
Efficiency									Not required.
HeatedPurge									Not required.
Inclusion									Not required.
InjectionVolume									Not required.
InjectionVolumeUnits									Not required.
InstrumentID	X	X	X	X	X	X	X	X	Report the laboratory identifier for the instrument used for this analysis.
LabAnalysisID	X	X	X	X	X	X	X	X	Report a unique identifier.
LabFileID	X	X	X	X	X	X	X	X	Report the lab file ID.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X	X	Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X	X	Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X	X	Report the month and year the SOW was issued.
PreparationBatch									Not required.
ProcedureID									Not required.
ProcedureName									Not required.
ReferenceDate									Not required.
ResultBasis	X	X	X		X				Report "Dry" for soil/sediment samples. For aqueous/water samples, report "Dissolved" if field-filtered; otherwise report "Total". Report "Wet" for tissue samples or for any other matrices (not aqueous/water) for which the results are not corrected for percent solids.
Temperature									Not required.
TemperatureUnits									Not required.
Wavelength									Not required.
WavelengthUnits									Not required.
Yield									Not required.
AnalysisGroup									Not required.
Handling									Not required.
ReportedResult	X	X	X	X	X	X	X		
AnalysisGroupID									Not required.
AnalyteGroupID	X	X	X	X	X	X	X		Report the unique identifier from the AnalyteGroup the Hardness result is derived from.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness or "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS analyses. Report "Derived" for Hardness.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Numbers as they appear in the SOW.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
ClientDetectionLimit									Not required.
ClientDetectionLimitUnits									Not required.
ClientQuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
ClientQuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
Comment									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the current MDL, adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures. Not required for Hardness.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL_sa".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.
ExpectedResult		X		X		X			Report the theoretical final calculated concentration (the spike added) for the spiked analytes or the true value for LCS.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits		X		X		X			Report "mg/kg" for soil/sediment, "ug/L" for aqueous/water (or mg/L for TCLP), or "ug" for wipe samples.
LabAnalysisID	X	X	X	X	X	X	X		Report the unique identifier from the analysis this reported result was derived from. Not required for Hardness.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report flags as specified in the SOW. Includes the Q qualifiers from Form 1-IN.
LabResultStatus	X	X	X						Report "Preliminary" or "Final" as applicable.
PeakID									Not required.
PercentDifference							X		Report the Percent Difference to the nearest whole percent.
PercentDifferenceLimitHigh							X		Report the upper limit for the Percent Difference to the nearest whole percent.
PercentDifferenceLimitLow									Not required.
PercentDifferenceLimitType							X		Report "Method".
PercentRecovery		X		X		X			Report the Percent Recovery.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
PercentRecoveryLimitHigh		X		X					Report the upper limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitLow		X		X					Report the lower limit for the Percent Recovery to the nearest whole percent.
PercentRecoveryLimitType		X		X					Report "Method".
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL adjusted for sample weight/volume, percent solids, and dilution factor to at least two significant figures.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL_sa".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		Report the final calculated result for detects.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for Hardness or TCLP) for aqueous/water, or "ug" for wipe samples.
RetentionTime									Not required.
RetentionTimeUnits									Not required.
RPD			X						Report the RPD to the nearest whole percent.
RPDLimitHigh			X						Report the upper limit for the RPD to the nearest whole percent.
RPDLimitType			X						Report "Method".
RPDType									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
PreparationPlusCleanup	X	X	X	X	X	X	X		
AliquotAmount	X	X	X	X	X	X	X		Report the sample amount in grams for soil/sediment or mL for aqueous/water to at least three significant figures. Not required for wipes.
AliquotAmountUnits	X	X	X	X	X	X	X		Report "g" for soil/sediment or "mL" for aqueous/water. Not required for wipes.
Analyst	X	X	X	X	X	X	X		Report the Analyst's initials.
CleanedUpDate									Not required.
CleanUpBatch									Not required.
CleanUpType									Not required.
ClientMethodCode									Not required.
ClientMethodID	X	X	X	X	X	X	X		Report the sample preparation ID as given in Exhibit B - Reporting and Deliverables Requirements.
ClientMethodModificationDescription									Not required.
ClientMethodModificationID									Not required.
ClientMethodName									Not required.
ClientMethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
ClientMethodVersion	X	X	X	X	X	X	X		Report the month and year the SOW was issued.
Comment									Not required.
FinalAmount	X	X	X	X	X	X	X		Report the volume of digestate produced by the preparation method in mL.
FinalAmountUnits	X	X	X	X	X	X	X		Report "mL".
InitialAmount									Not required.
InitialAmountUnits									Not required.
LabID									Not required.
LabMethodID									Not required.
LabMethodName									Not required.
LabName									Not required.
LotNumber									Not required.
MethodCode									Not required.
MethodID	X	X	X	X	X	X	X		Report "ISM02.4".
MethodModificationDescription									Not required.
MethodModificationID									Not required.
MethodName									Not required.
MethodSource	X	X	X	X	X	X	X		Report "EPA_CLP".
MethodVersion	X	X	X	X	X	X	X		Report the month and year the SOW was issued.
PreparationBatch	X	X	X	X	X	X	X		Links all samples that were prepared together. Report a unique identifier for each batch.
PreparationPlusCleanupType	X	X	X	X	X	X	X		Report "Preparation".
PreparationType	X	X	X	X	X	X	X		Report "Automated" or "Manual".
PreparedDate	X	X	X	X	X	X	X		Report the date and time the sample was prepared.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
ProcedureID									Not required.
ProcedureName									Not required.
Solvent									Not required.
Analyte	X	X	X	X	X	X	X		
AnalyteGroupID	X	X	X	X	X	X	X		Report the identifier that links the Ca or Mg result to the AnalyteGroup Hardness result.
AnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Target" for all target analytes except Hardness; "Spike" for all target analytes designated as spike analytes for Matrix Spike, Post-Digestion Spike, and LCS; "Internal_Standard" for internal standards; or "Monitor" for non-target interferences and masses requiring monitoring.
BiasErrorRatio									Not required.
CASRegistryNumber	X	X	X	X	X	X	X		Report the CAS Number as it appears in the SOW.
ClientAnalyteID	X	X	X	X	X	X	X		Report CAS number.
ClientAnalyteName	X	X	X	X	X	X	X		Report the analytes as they appear in the SOW.
Comment									Not required.
Counts									Not required.
CountsUncertainty									Not required.
CountsUncertaintyConfidenceLevel									Not required.
CountsUncertaintyDetermination									Not required.
CountsUncertaintyIntervalType									Not required.
CountsUncertaintyLimitHigh									Not required.
CountsUncertaintyLimitLow									Not required.
CountsUncertaintyType									Not required.
CountsUnits									Not required.
DetectionLimit	X	X	X	X	X	X	X		Report the MDL to at least two significant figures.
DetectionLimitType	X	X	X	X	X	X	X		Report "MDL".
DetectionLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
DifferenceErrorRatio									Not required.
Efficiency									Not required.
ExpectedResult									Not required.
ExpectedResultUncertainty									Not required.
ExpectedResultUncertaintyConfidenceLevel									Not required.
ExpectedResultUncertaintyDetermination									Not required.
ExpectedResultUncertaintyIntervalType									Not required.
ExpectedResultUncertaintyLimitHigh									Not required.

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
					Applicability				
ExpectedResultUncertaintyLimitLow									Not required.
ExpectedResultUncertaintyType									Not required.
ExpectedResultUncertaintyUnits									Not required.
ExpectedResultUnits									Not required.
Inclusion									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW.
LotNumber	X	X	X	X	X	X	X		Report the vendor/manufacturer-assigned lot number for this standard (Internal Standards and spiking analytes only).
PeakID	X	X	X	X	X	X	X		If response from a single peak is used for quantitation, report the ID of that peak.
PercentRecovery									Not required.
PercentRecoveryLimitHigh									Not required.
PercentRecoveryLimitLow									Not required.
PercentRecoveryLimitType									Not required.
PercentRecoveryType									Not required.
QuantitationLimit	X	X	X	X	X	X	X		Report the CRQL.
QuantitationLimitType	X	X	X	X	X	X	X		Report "CRQL".
QuantitationLimitUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
ReportingLimit									Not required.
ReportingLimitType									Not required.
ReportingLimitUnits									Not required.
Result	X	X	X	X	X	X	X		For target or spike analyte detects, and for monitored masses, report the final calculated result.
ResultLimitHigh									Not required.
ResultLimitLow									Not required.
ResultLimitType									Not required.
ResultType	X	X	X	X	X	X	X		Report "=" for all detected analytes. Report "Not_Detected" for non-detects. Report "Negative" for PB or LEB results less than the negative MDL (-MDL).
ResultUncertainty									Not required.
ResultUncertaintyConfidenceLevel									Not required.
ResultUncertaintyDetermination									Not required.
ResultUncertaintyIntervalType									Not required.
ResultUncertaintyLimitHigh									Not required.
ResultUncertaintyLimitLow									Not required.
ResultUncertaintyType									Not required.
ResultUncertaintyUnits									Not required.

Sample

Node and Data Elements	Sample	MS	Dup	LCS	PB/LEB	PDS	SD	NCS	Instructions
ResultUnits	X	X	X	X	X	X	X		Report "mg/kg" for soil/sediment, "ug/L" (or "mg/L" for TCLP) for aqueous/water, or "ug" for wipe samples.
StandardSource	X	X	X	X	X	X	X		Report the vendor/manufacturer for this standard.
Wavelength									Not required.
WavelengthUnits									Not required.
AnalyteGroup	X	X	X	X	X	X	X		
AnalyteGroupID	X	X	X	X	X	X	X		Report a unique identifier.
AnalyteName	X	X	X	X	X	X	X		Report "Hardness".
AnalyteNameContext	X	X	X	X	X	X	X		Report "CAS".
AnalyteType	X	X	X	X	X	X	X		Report "Derived".
CASRegistryNumber	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteID	X	X	X	X	X	X	X		Report "Hardness".
ClientAnalyteName	X	X	X	X	X	X	X		Report "Hardness".
Comment									Not required.
LabAnalyteID									Not required.
LabQualifiers	X	X	X	X	X	X	X		Report qualifiers as specified in the SOW. Include the Q qualifiers from Form 1-IN.
Result	X	X	X	X	X	X	X		Report the final calculated for detects per the SOW.
ResultType	X	X	X	X	X	X	X		Report "=" for detects. Report "Not_Detected" for non-detects (where both Ca and Mg are not detected).
ResultUncertainty									Not required.
ResultUnits	X	X	X	X	X	X	X		Report "mg/L".

TABLE 4. ABBREVIATIONS

Abbreviation/Acronym	Definition
%R	Percent Recovery
%RSD	Percent Relative Standard Deviation
CAS	Chemical Abstracts Service
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CN	Cyanide
CRQL	Contract Required Quantitation Limit
DTD	Document Type Definition
Dup	Duplicate Sample
EDD	Electronic Data Deliverable
Hg	Mercury
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICS	Interference Check Sample
ICSA	Interference Check Sample Solution A
ICSAB	Interference Check Sample Solution AB
ICV	Initial Calibration Verification
ID	Identifier
IEC	Interelement Correction
IPC	Instrument Performance Check (Tune)
Lab	Laboratory
LCS	Laboratory Control Sample
LEB	Leachate Extraction Blank
MDL	Method Detection Limit
MS	Matrix Spike
NCS	Non-Client (ZZZZZZ) Sample
PB	Preparation Blank
PDS	Post-Digestion/Distillation Spike
QATS	Quality Assurance Technical Support
QC	Quality Control
%RI	Percent Relative Intensity
RPD	Relative Percent Difference
SD	Serial Dilution
SDG	Sample Delivery Group
SPLP	Synthetic Precipitation Leaching Procedure
SOW	Statement of Work
TCLP	Toxicity Characteristic Leaching Procedure
u	Atomic Mass Unit

APPENDIX A – FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

1.0   FORMAT CHARACTERISTICS FOR METHOD DETECTION LIMIT STUDY DATA

The Method Detection Limit (MDL) study data deliverable consists of a Microsoft® Excel spreadsheet containing the following columns (Table A-1) in the order specified.

The "Required" field in Table A-1 identifies the columns that are always required to be populated.

The Contractor shall provide one spreadsheet for each combination of instrument ID, analytical method, and preparation method used to report results under this contract.

The Contractor shall deliver the spreadsheets to the recipients specified in Table 1 of Exhibit B – Reporting and Deliverables Requirements.

The format for the Microsoft® Excel file name shall be MDL_#.xls, where # can be any naming convention selected by the Contractor.

TABLE A-1. MDL STUDY DATA DELIVERABLE

Column	Required	Instruction
LabID	X	Report the agency-assigned Lab Code.
LabContract	X	Report the Lab Contract Number per the instructions for Header/LabContract.
MethodSource	X	Report the SOW per the instructions for SamplePlusMethod/ClientMethodID.
Method	X	Report the analytical method per the instructions for Header/LabDataPackageName.
PreparationMethod	X	Report the preparation method per the instructions for PreparationPlusCleanup/ClientMethodID.
ClientMethodCategory		Leave null.
ClientMethodModificationID		Report the MA number per the instructions for SamplePlusMethod/ClientMethodModificationID if applicable. Otherwise leave null.
Level		Leave null.
Matrix	X	Report the sample matrix per the instructions for SamplePlusMethod/MatrixID.
InstrumentID	X	Report the instrument ID per the instructions for Analysis/InstrumentID.
ColumnID		Leave null.
ClientAnalyteID	X	Report the analyte per the instructions for ReportedResult/ClientAnalyteID.
Peak	X	Report the wavelength or mass at which measurements are taken per the instructions for Peak/Wavelength or Peak/Mass.
ResultUnits	X	Report the units for the replicate concentrations reported per the instructions for ReportedResult/ResultUnits.

Column	Required	Instruction
Replicate##	X	The Laboratory shall include as many columns as there are replicates reported. Usually this would be seven, but more than seven replicates can be reported. The Laboratory shall report the results of the analysis of each replicate for each analyte. Each column shall be labeled "Replicate##", where the ## shall be replaced with the numeric designation of the replicate (e.g., Replicate01 for the first, Replicate02 for the second, Replicate03 for the third, etc.).
LabAnalysisID##	X	Following each Replicate## column, the Laboratory shall report a LabAnalysisID## column, reporting the LabAnalysisID of that replicate for that analyte per the instructions for Analysis/LabAnalysisID. The LabAnalysisID## columns shall be labeled in the same manner as the Replicate## columns.
AnalyzedDate##	X	Following each LabAnalysisID## column, the Laboratory shall report a AnalyzedDate## column, reporting the analysis date and time for that replicate for that analyte per the instructions for the Analysis/AnalyzedDate data element. The AnalyzedDate## columns shall be labeled in the same manner as the Replicate## columns. (MMDDYYYYThh:mm:ss)
StandardDeviation	X	Report the calculated standard deviation of the replicates for each analyte to at least three significant figures.
StudentsTValue	X	Report the appropriate Student's T value for the degrees of freedom based on the number of replicates and 99% for the one-sided test.
DetectionLimit	X	Report the calculated Detection Limit for each analyte per the instructions for Analysis/Analyte/DetectionLimit.
DetectionLimitUnits	X	Report the appropriate units for the preparation method per the instructions for Analysis/Analyte/DetectionLimitUnits.
MDLAcceptable	X	Enter "Y" if the calculated MDL is less than one-half the CRQL for the analyte and matrix. Otherwise enter "N".
ExpectedResult	X	Report the concentration for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResult.
ExpectedResultUnits	X	Report the concentration units for each analyte in the MDL standards per the instructions for ReportedResult/ExpectedResultUnits.

Column	Required	Instruction
ConcentrationAcceptable	X	Enter "Y" if the concentration of the analyte in the MDL standards was less than or equal to 10 times the calculated MDL for that analyte. Otherwise enter "N".
EffectiveDate	X	Report the date on which the Laboratory began to use the calculated MDL for reporting sample results for that analyte, instrument, and method formatted per the instructions for Header/DateFormat. This date cannot precede the analysis date of the MDL replicates.

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